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Civil District Court

Parish of Orleans

State of Louisiana

No. 96-8461 Division "K"

----------X

Gloria Scott, Et Al

versus

The American Tobacco Company, Inc., Et Al

----------X

Videotaped deposition of

Harold Burnley, Jr.

taken on

October 10, 2000

951 East Byrd Street

Richmond, Virginia

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* * *

VIDEOGRAPHER: Today is the 10th of October, 2000. This is videotape deposition of Harold Burnley. We are going on the record at 9:14 A.M.

* * *

HAROLD BURNLEY,

having been first duly sworn, was examined and testified as follows:

* * *

EXAMINATION

* * *

BY MR. HOPPER:

Q. Good morning, Mr. Burnley.

A. Good morning.

Q. My name is Randy Hopper and I represent the plaintiffs in this action against your employer, Philip Morris.

I think that for ease and comfort for all, I'm going to take care of a few housekeeper matters right up front with your counsel so that we can kind of speed the deposition along as best we can.

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09:16:02 One of the housekeeping
09:16:03 matters that I want to address
09:16:05 initially is that in the questioning
09:16:08 and the answer give and take, you have
09:16:10 been deposed many times, I am aware,
09:16:21 and I think you are familiar with this
09:16:21 process. But just as a gentle
09:16:[illegible] reminder, please let me finish all the
09:16:25 questions before you try to answer and
09:16:25 I'll try my best to return that
09:16:27 courtesy, to allow you to finish all
09:16:[illegible] your answers before I ask another
09:16:[illegible] question.
09:16:30 You are also aware that
09:16:32 counsel in a deposition have the right
09:16:34 to object. Your counsel has the right
09:16:39 to object. I have the right to object
09:16:[illegible] and we have the opportunity, according
09:16:[illegible] to the procedure in a deposition, to
09:16:[illegible] enter those objections into the record.
09:16:55 And counsel and I, in our
09:16:55 experience will do our best, I'm sure,
09:16:55 to give each other the time and the
09:16:55 room to make those objections so that
09:16:59 again, it creates ease and it creates a

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09:17:03 procedure that is favorable to
09:17:07 everyone, particularly the Court
09:17:09 Reporter.
09:17:10 One of the things we want to
09:17:12 do to begin with is to enter into the
09:17:16 record a number of stipulations.
09:17:18 Counsel and I, Mr. Muehlberger, have
09:17:28 essentially agreed to these and I would
09:17:30 like to get those on the record.
09:17:32 I don't know if you want to
09:17:33 pan on this or not or we'll just do it
09:17:33 by audio on this one.
09:17:35 That the deposition is being
09:17:43 conducted by the agreement of the
09:17:43 parties; that any defects with respect
09:17:44 to notice, time and location are
09:17:46 waived; that any portion of the
09:17:48 deposition can be used for any purpose
09:17:50 by any party at trial; the deposition
09:17:53 shall be on the record at all times;
09:17:59 one defense attorney may object on
09:18:00 behalf of the deponent; plaintiffs can
09:18:03 utilize up to two attorneys to question
09:18:07 the witness, as long as questioning is
09:18:10 not repetitive; and the witness may not

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09:18:13 confer with the defense counsel until
09:18:16 completion of the deposition.
09:18:19 So, having so entered those
09:18:27 stipulations on the record, we would
09:18:27 now like to enter just a few other
09:18:34 items for housekeeping purposes, which
09:18:38 would be to enter the CMO-2 signed by
09:18:43 the Court, and the deposition notice in
09:18:46 the case.
09:18:48 Take a quick look at that.
09:19:06 And then, for the record --
09:19:14 MR. MUEHLBERGER: Randy, you
09:19:15 have some others things attached
09:19:17 here.
MR. HOPPER: Yes, I was just
coming to that, so if you want to
hang onto it for just a minute.
(Whereupon, the deposition
notice and the CMO-2 was marked
Burnley Exhibit No. 1 for
Identification.)
09:19:20 We want to enter into the
09:19:31 record any reliance material that
09:19:31 the witness has brought to the
09:19:31 deposition or has provided prior to

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09:19:31 the deposition.

09:19:31 And have both this CMO-2 and

09:19:33 this reliance material marked.

MR. HOPPER: Jim, have we been served with these other materials? I know we have this. It was incorporated by reference into his -- with his C.V. Report.

MR. MUEHLBERGER: My recollection, Randy, is that the reliance list was actually beneath the citation of authority. And what I think you'll find this design is, is his publication.

09:20:21 MR. HOPPER: I'm not going to

09:20:25 take the time to read all these,

09:20:27 but for the Court Reporter's

09:20:28 purposes, in addition to marking

09:20:28 the CMO-2 as an exhibit -- the

09:20:36 notice as Exhibit Number 1 and the

09:20:36 CMO-2 as Exhibit Number 2.

09:20:36 And I think -- why don't we

09:20:36 call these Burnley Number 1, ad

09:21:09 infinitum.

09:21:09 That's the notice, I believe,

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as well as the CMO-2.

And then, I'm entering into the record Burnley Exhibit Number 2, which are the reliance documents provided by the witness, which comprises those documents and the material that he is relying on for the basis of his expert testimony today.

(Whereupon, the reliance documents were marked as Exhibit No. 2 for Identification)

BY MR. HOPPER:

Q. Are there any fee agreements or any fee statements or time records or anything to this point that you would provide me, Mr. Burnley?

A. No.

MR. HOPPER: If, in fact, and I'm not privilege to this information, but in the event that it is discoverable, I simply want to place on the record an objection -- excuse me -- place on the record the right to reserve the

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09:22:32 opportunity to reopen the
09:22:34 deposition at some point if, in
09:22:38 fact, that becomes an issue or a
09:22:39 matter in the case.
09:22:41 And then I think we may have
09:22:44 this when we introduce
09:22:49 Mr. Burnley's C.V., because I
09:22:[illegible] believe you're saying this -- as I
09:22:56 recall, this is a paper that he
09:22:57 authored that was included along
09:23:01 with his C.V.?
09:23:[illegible] MR. MUEHLBERGER: That's
09:23:[illegible] right.
09:23:05 MR. HOPPER: So, I think we'll
09:23:05 just treat that at that time, Jim.
09:23:05 MR. MUEHLBERGER: Yes, the
09:23:05 reliance list is the page right
09:23:05 here.
09:23:[illegible] MR. HOPPER: Okay.
09:24:[illegible] BY MR. HOPPER:
09:24:23 Q. And just a further -- a few
09:24:27 housekeeping matters, Mr. Burnley,
09:24:30 before we get into some of the
09:24:32 substance.
09:24:33 I would like to remind you, if

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09:24:36 your counsel hasn't, or to follow up,
09:24:42 that this deposition is not only being
09:24:42 taken for discovery purposes but is
09:24:53 also a trial deposition and will be
09:24:53 used at trial and that you are aware of
09:24:53 that. And that, in effect, you are
09:24:53 testifying to the jury when you provide
09:24:56 testimony today, is that understandable
09:24:58 to you?
09:24:59 A. Yes.
09:25:01 Q. I would also like to remind
09:25:02 you that this deposition is being taken
09:25:06 on behalf of the citizens of Louisiana,
09:25:08 the State of Louisiana and that, in
09:25:10 effect, they are our clients,
09:25:14 represented by these class
09:25:17 representatives; is that clear to you?
09:25:19 A. Yes.
09:25:21 MR. MUEHLBERGER: You may have
09:25:30 said State of Louisiana. You mean
09:25:33 citizens of the State of Louisiana.
09:25:46 MR. Hopper: Yes. If I
09:25:46 misspoke, I apologize.
09:25:48 I believe that now covers all
09:25:49 of the housekeeping procedures that I

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09:25:55 have at this point.

09:25:56 Counsel, was there anything

09:25:58 you needed or wanted to address at this

09:26:01 early stage.

09:26:02 MR. MUEHLBERGER: No.

09:26:02 BY MR. HOPPER:

09:26:02 Q. Okay, we'll proceed with the

09:26:05 substance of the deposition now at this

09:26:12 point.

09:26:12 Mr. Burnley, you understand

09:26:15 that your testimony is provided under

09:26:18 oath today and having sat for

09:26:18 depositions in the past, you know that

09:26:20 all of your testimony must be true to

09:26:24 the best of your knowledge and

09:26:27 information?

09:26:30 A. Yes, I do.

09:26:31 Q. You are aware of the fact that

09:26:37 because this is a deposition taken both

09:26:37 for purposes of a fact deposition as

09:26:40 well as for trial, that when you're

09:26:43 addressing my questions, that you are,

09:26:45 in effect, addressing the Jury?

09:26:48 A. I understand.

09:26:51 Q. Okay. In preparation for your

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09:26:53 deposition today, Mr. Burnley, did you
09:26:56 meet with anyone before coming here
09:26:59 this morning?
09:27:01 A. I did. I met yesterday for
09:27:03 about an hour-and-a-half with Mr.
09:27:07 Muehlberger, Ms. Brown and Mr. Webb, I
09:27:10 believe.
09:27:10 Q. And did you meet with any
09:27:12 other -- and they are your counsel for
09:27:16 Philip Morris, correct?
09:27:17 A. Yes.
09:27:20 Q. And did you meet with any
09:27:20 other attorneys who represent Philip
09:27:21 Morris in this proceeding in
09:27:25 preparation --
09:27:26 A. No.
09:27:27 Q. In the past, have you met with
09:27:31 any other attorneys or any in-house
09:27:34 counsel for Philip Morris in
09:27:36 preparation for this deposition?
09:27:37 A. No.
09:27:38 Q. Have you met with any
09:27:41 colleagues or any other employees of
09:27:42 Philip Morris in preparation for this
09:27:45 deposition?

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09:27:50 A. No.

09:27:50 Q. Have you met with any other expert witnesses or conferred with any of the other expert witnesses that your company has disclosed in this proceeding?

09:27:59

09:28:00 A. No.

09:28:00 Q. Have you reviewed any of the material presented -- any of the reliance documents by any of those other expert witnesses?

09:28:00

09:28:04

09:28:04 A. No.

09:28:08 Q. Can you tell me what material you have reviewed in preparation for this deposition?

09:28:11

09:28:12 A. I actually have not reviewed any material in preparation for this deposition.

09:28:14

09:28:17

09:28:18 Q. All right. Just so we are clear, I want to address that in particular.

09:28:19

09:28:25 You have not gone back and reviewed any of your prior testimony from the Small lawsuit in New York?

09:28:25

09:28:25

09:28:26 A. No, I have not.

09:28:30

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09:28:32 Q. Or the Barnes proceeding in
09:28:39 Pennsylvania?
09:28:39 A. No.
09:28:39 Q. Or the Engle proceeding or
09:28:40 trial in Florida?
09:28:42 A. No.
09:28:46 Q. Or the ABC lawsuit?
09:28:49 A. No.
09:28:50 Q. Okay. You would just as soon
09:28:52 forget about those, right?
09:28:55 Have you reviewed any
09:28:58 materials, either that you had in your
09:29:03 own possession or provided to you from
09:29:06 your employer or anyone who works at
09:29:09 Philip Morris that relates to the
09:29:12 substance of this lawsuit?
09:29:14 MR. MUEHLBERGER: In
09:29:14 preparation for the deposition?
09:29:14 BY MR. HOPPER:
09:29:15 Q. In preparation. All of these
09:29:18 are prefaced by that, certainly.
09:29:22 A. I have not.
09:29:25 Q. When you met with Mr.
09:29:30 Muehlberger yesterday, how long did you
09:29:31 say you met with him?

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09:29:33 A. About an hour-and-a-half.
09:29:36 Q. Did you talk with him by
09:29:39 telephone prior to the deposition to
09:29:40 prepare for this deposition today?
09:29:43 A. No.
09:29:49 Q. And you haven't conferred with
09:29:49 any of the other officers or any other
09:29:50 colleagues of Philip Morris then?
09:29:54 MR. MUEHLBERGER: Asked and
09:29:54 answered.
09:29:56 A. No, I haven't.
09:29:56 BY MR. HOPPER:
09:30:05 Q. You may notice, I dispensed
09:30:07 with some of the early formalities of
09:30:10 having you state your name for the
09:30:13 record, because I think we have covered
09:30:17 so much of that in other depositions in
09:30:20 the past that I don't want to take the
09:30:23 time to go into that.
09:30:24 Can you state your current job
09:30:26 for the record, and title, please.
09:30:29 A. Yes, I'm Vice-President of
09:30:37 Operations Planning for Philip Morris,
09:30:37 USA.
09:30:37 Q. And as I see from your C.V.,

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09:30:39	1	you have held that position since on or
09:30:42	2	about -- in or about May of 1998?
09:30:46	3	A. That's correct.
09:30:47	4	Q. And prior to that, you held a
09:30:52	5	number of different jobs that I believe
09:30:52	6	span, over the course of time, dating
09:30:54	7	back to 1974 with Philip Morris?
09:30:58	8	A. 1973.
09:31:00	9	Q. So you came in '73 from Allied
09:31:04	10	Chemical?
09:31:05	11	A. That's right.
09:31:11	12	Q. I also notice from your C.V.
09:31:11	13	that in 1967, you received a degree, a
09:31:14	14	Bachelor's of Science degree in
09:31:16	15	Chemical Engineering from Virginia
09:31:19	16	Tech; is that correct?
09:31:20	17	A. Yes, it is.
09:31:24	18	Q. And I see you grew up with
09:31:26	19	Frank Sinatra in Hoboken?
09:31:31	20	A. Well, I didn't grow up with
09:31:36	21	him but I did go to Stevens for a
09:31:42	22	while, yes.
09:31:51	23	Q. Can you describe for me and
09:31:55	24	for the Jury, the responsibilities that
09:32:08	25	are involved in your job in Operations

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09:32:08 Planning. And if you wouldn't mind, to
09:32:08 go a step further with Process
09:32:08 Development, since that's a division, I
09:32:08 believe, of the R & D Department at
09:32:11 Philip Morris?
09:32:11 A. Sure. My job as
09:32:12 Vice-President of Operations Planning
09:32:14 is really to make sure that Philip
09:32:19 Morris, USA has a strategy and a set of
09:32:24 operational plans that are consistent
09:32:29 with the mission of the company. And a
09:32:32 set of plans that will enable the
09:32:33 integration of the various departments
09:32:37 in Philip Morris to collaborate to
09:32:49 deliver against those strategies.
09:32:49 I also have responsibility for
09:32:49 production planning and production
09:32:49 scheduling.
09:32:49 As VP of Process Development
09:32:52 in R & D, I was really responsible for
09:33:02 defining and developing the tobacco
09:33:02 processing technologies that the
09:33:03 company needed strategically to meet
09:33:07 its long-term objectives.
09:33:10 We also did quite a bit of

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09:33:12 work in trying to optimize, from a
09:33:12 product quality or a product cost point
09:33:17 of view, the manufacturing processes
09:33:19 that were already in use.
09:33:23 Q. In past testimony, I'm aware
09:33:28 that you have, in one way or another,
09:33:32 distinguished your job from operations.
09:33:38 Do you consider now that you are a
09:33:41 Vice-President of Operations Planning
09:33:44 that you are very much involved with
09:33:46 the operations of Philip Morris?
09:33:47 A. Oh, absolutely.
09:33:53 Q. And do you report now to the
09:33:53 COO of Philip Morris?
09:33:53 A. I report to Dave Beran, who is
09:33:58 Senior Vice-President of Operations.
09:34:06 Q. Would you spell that for me,
09:34:06 please, the last name.
09:34:09 A. B-E-R-A-N.
09:34:10 Q. Now, at one point in the past,
09:34:13 you reported to Ms. Cathy Ellis?
09:34:17 A. Yes.
09:34:17 Q. And she was the Senior
09:34:19 Vice-President of R & D at Philip
09:34:25 Morris; is that correct?

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09:34:25 A. That's correct.
09:34:25 Q. Does she still hold that
09:34:26 position?
09:34:26 A. She does not.
09:34:26 Q. Who is in charge of R & D at
09:34:30 this time?
09:34:30 A. Urs Nyffeler.
09:34:30 Q. Would you spell that for me.
09:34:38 A. U-R-S N-Y-F-F-E-L-E-R.
09:35:06 Q. Are you -- did someone replace
09:35:07 you as Vice-President of Process
09:35:10 Development when you became
09:35:13 Vice-President of Operations and
09:35:14 Planning?
09:35:15 A. Not exactly. There was a
09:35:17 fairly massive reorganization and
09:35:21 restructuring in Philip Morris, USA,
09:35:25 and my job was sort of broken up and
09:35:30 parceled out to a number of different
09:35:33 departments. So, it's pretty hard to
09:35:33 match some of the old jobs with the
09:35:35 current structure.
09:35:36 Q. Are you still involved with
09:35:38 Process Development?
09:35:47 A. Yes. In fact, one of my

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09:35:47 accountabilities is to do planning for
09:35:47 the research, development and
09:35:49 engineering organization.
09:35:55 Q. I'm aware, and you may have to
09:35:58 correct me on this, that R & D at
09:36:03 Philip Morris was separated at one time
09:36:04 into three departments -- process
09:36:07 development, scientific affairs and
09:36:09 then I'm not sure what the third
09:36:12 department was. Was that the leaf
09:36:15 department?
09:36:15 A. No. There have been a number
09:36:16 of structures over the years but
09:36:16 generally the partitioning was product
09:36:21 development, process development,
09:36:24 research; and there was a scientific,
09:36:27 later a scientific affairs group or
09:36:28 organization as well.
09:36:29 Q. And that is what Dr. Carchman
09:36:32 headed?
09:36:32 A. Yes.
09:36:33 Q. Does he still head that?
09:36:35 A. No, he does not.
09:36:37 Q. Who heads that division now?
09:36:46 A. Rick Solana.

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09:36:46 Q. Do you distinguish your job as
09:36:48 Vice-President of Operations Planning
09:36:50 from the manufacturing position, or do
09:36:55 you consider yourself a part of the
09:36:55 manufacturing at Philip Morris?
09:36:57 A. Well, I'm part of the
09:36:57 Operations Organization, which includes
09:36:59 Manufacturing, Leaf, Purchasing,
09:37:06 Research Development and Engineering,
09:37:08 Quality Systems.
09:37:08 And I'm probably forgetting a
09:37:12 couple of other departments, but those
09:37:18 are the main departments. So, my job
09:37:18 is make sure we have got an integrated
09:37:21 long-term strategy and plan to meet out
09:37:24 objectives that crosses all of those
09:37:27 departments.
09:37:42 Q. And does Mr. Beran report to
09:37:44 the CEO or President of Philip Morris
09:37:46 then?
09:37:49 A. He does.
09:37:49 Q. And is that Mr. Szymanczyk
09:37:55 now?
09:37:55 A. That's correct.
09:38:04 Q. At one time you provided

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09:38:06 testimony in one of the other cases I
09:38:09 referenced earlier, that you had no
09:38:11 direct accountability for Operations at
09:38:19 Philip Morris.
09:38:19 Do you consider that you do
09:38:19 have direct accountability for
09:38:19 Operations at Philip Morris now, with
09:38:21 this new job since May of 1998?
09:38:26 MR. MUEHLBERGER: Just object
09:38:26 to the extent it misstates prior
09:38:29 testimony.
09:38:31 A. I guess I would have to go
09:38:32 back and read the testimony.
09:38:32 But what I think I said, if I
09:38:34 recall correctly is, I was not part of
09:38:36 the Manufacturing Department.
09:38:37 Operations is a broader
09:38:41 accountability than just manufacturing.
09:38:41 I was not part of the
09:38:47 Manufacturing Department, I'm still not
09:38:49 part of the Manufacturing Department
09:38:50 but I'm part of the Operations, which
09:38:53 includes manufacturing.
09:38:55 If that helps clears it up.
09:38:55 BY MR. HOPPER:

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09:38:56 Q. Well, is it more a corporate
09:38:59 level type job then, as opposed to a
09:39:02 line job?
09:39:02 A. It is, yes.
09:39:11 Q. Okay. At one point I believe
09:39:12 in testimony you have also provided,
09:39:16 that you said there were approximately
09:39:18 one hundred fifty people that reported
09:39:20 to you.
09:39:23 Do you still have that number
09:39:24 of people reporting to you?
09:39:25 A. No, I think in my current job
09:39:29 I have fifty-five or so.
09:39:41 Q. Can you identify on your C.V.
09:40:03 which job you held where you reported
09:40:08 to Mr. Ken Houghton at one time?
09:40:09 A. Sure. Actually, two.
09:40:28 Director of Process Development and
09:40:41 Vice-President of Process Development.
09:40:40 Q. Is Dr. Cathy Ellis still with
09:40:46 Philip Morris?
09:40:48 A. No, she's not.
09:40:48 Q. She retired?
09:40:49 A. She left the company, yes.
09:40:56 I'm not sure whether she actually

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09:40:56 retired or not.

09:41:34 Q. In your current job as Vice-President of Operations Planning, do you still get involved in the engineering aspect of the Process Department the way you did in the past?

09:41:47 A. Well, I don't have direct accountability to run the Process Department organization today, but I'm still very interested in it and yeah, I'm still part of it in my planning role.

09:42:00 Q. Well, in high school and at Virginia Tech in preparation for your Degree in Chemical Engineering, I would imagine you've studied a considerable amount of chemistry?

09:42:39 A. Yes.

09:42:40 Q. And have you done any advanced work or additional programs related to your Degree or your profession?

09:42:48 A. Yes, I was actually enrolled in a Master's Program at Stevens.

09:42:55 After graduation I went to work for Dupont in New Jersey and

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09:43:03 entered Stevens as an evening student
09:43:11 and was majoring in chemical
09:43:11 engineering. And returned a couple of
09:43:11 years later to the Richmond, Virginia,
09:43:13 area and transferred to Virginia Tech.
09:43:16 They had a local extension school here
09:43:23 and I continued graduate studies in
09:43:24 Chemical Engineering at Virginia Tech.
09:43:28 Q. That is what you refer to on
09:43:30 your C.V. as post-graduate studies in
09:43:47 Chemical Engineering?
09:43:41 A. That's correct.
09:43:[illegible] Q. Are you licensed as an
09:43:46 engineer?
09:43:46 A. I am.
09:43:[illegible] Q. Under the auspices of the
09:43:48 State of Virginia?
09:43:51 A. Yes.
09:43:[illegible] Q. And what procedures did you
09:43:[illegible] have to go through in order to become
09:43:54 licensed as an engineer?
09:43:58 A. Had to sit for a two-day exam.
09:44:00 Q. Do you recall what year that
09:44:01 was? Was it right out of college?
09:44:04 A. No, it was -- I can give you

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09:44:06 an approximate year. It was around
09:44:06 1973.
09:44:14 Q. Was that certification and
09:44:16 licensure specific to chemical
09:44:19 engineering or across the board for
09:44:22 engineering?
09:44:25 A. It was across the board for
09:44:25 engineering.
09:44:26 Q. So, theoretically, you could
09:44:28 be licensed to work as an engineer in
09:44:33 mechanical engineering or other areas
09:44:36 of engineering as well?
09:44:36 A. If I felt competent, yes.
09:44:38 Q. Have you confined most of your
09:44:41 career, whether with Dupont or Allied
09:44:44 or with Philip Morris, to chemical
09:44:47 engineering then?
09:44:49 A. Chemical engineering and
09:44:51 control systems engineering.
09:44:54 Q. What is control systems
09:44:56 engineering?
09:44:57 A. It's application of
09:44:58 electronics and computer systems to
09:45:02 automatically control industrial
09:45:04 processes.

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09:45:22 Q. I assume you were trained and
09:45:25 are -- you consider yourself
09:45:27 considerably computer literate then?
09:45:31 A. I hope so. Although it's
09:45:33 getting tougher.
09:45:36 Q. But you understand the
09:45:39 intricacies and the complexities
09:45:41 associated with computer automation of
09:45:46 industrial processes, then?
09:45:51 A. Yes.
09:45:51 Q. I assume as a system engineer,
09:45:52 when you first came to Philip Morris,
09:45:54 much of that job was involved in the
09:45:58 monitoring and the development of those
09:46:05 computerized automated systems that ran
09:46:05 the manufacturing?
09:46:05 A. That's exactly right.
09:46:06 Q. And for example, as chief
09:46:08 process engineer, you would have done
09:46:09 the same thing except you would have
09:46:12 had a lot of people helping you and
09:46:12 working for you, but you would have
09:46:14 rendered higher judgments than they on
09:46:17 those systems and processes?
09:46:19 A. That's true, although as Chief

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09:46:24 Process Engineer, I also had design
09:46:26 responsibility for the industrial
09:46:28 processes as well, as just the control
09:46:31 systems accountability.
09:46:48 Q. As an officer of the company
09:46:48 who is involved in strategic planning
09:46:50 and production planning and development
09:46:54 of procedures and processes for Philip
09:46:56 Morris, involving Manufacturing, Leaf
09:46:58 Purchasing, R & D, and Quality Control,
09:47:07 do you meet regularly with the heads of
09:47:07 those various divisions?
09:47:07 A. Yes.
09:47:08 Q. On what basis? How often?
09:47:09 A. At least twice a month.
09:47:12 Q. And are those set meetings on
09:47:16 your calendar?
09:47:16 A. They are.
09:47:17 Q. Standing, running meetings?
09:47:18 A. Yes.
09:47:59 Q. Now, Mr. Burnley, you have
09:48:01 been disclosed in this litigation as
09:48:04 both a fact and expert witness; is that
09:48:07 correct?
09:48:07 A. Yes.

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09:48:09 Q. And as an expert witness, how
09:48:12 would you define and what would you
09:48:14 consider to be the expertise you are
09:48:18 providing to Philip Morris, the
09:48:18 defendant in this case?
09:48:21 A. Well, my field is really how
09:48:25 the company makes its product. So, to
09:48:29 the extent to which I can answer
09:48:34 questions about any of our
09:48:34 manufacturing processes, to a certain
09:48:38 extent about ingredients and how
09:48:41 ingredients are used, those are
09:48:44 primarily the areas of my expert
09:48:48 testimony.
09:48:59 Q. And the statement of your
09:49:01 opinions that you have offered as an
09:49:06 expert in this litigation, states that
09:49:09 you would be prepared to offer
09:49:12 testimony as a mechanical and process
09:49:16 engineer in the following areas:
09:49:16 Manufacturing processes, including
09:49:17 process controls used in making
09:49:19 cigarettes; is that correct?
09:49:21 A. That's correct.
09:49:25 Q. Including state of the art in

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09:49:27 cigarette manufacture?
09:49:29 A. Yes.
09:49:32 Q. You are prepared to testify as
09:49:34 to the reasons why Philip Morris uses
09:49:36 ammonia compounds in its manufacturing
09:49:43 processes?
09:49:43 A. I am.
09:49:43 Q. You are prepared to testify
09:49:47 and offer opinions on the manufacture
09:49:48 of various tobacco blend components
09:49:54 used in the manufacture of commercial
09:49:54 cigarette?
09:49:54 A. Yes.
09:49:56 Q. Including reconstituted
09:49:59 tobacco processes?
09:49:59 A. Yes.
09:50:05 Q. And you are also prepared to
09:50:09 testify and offer opinions expressed by
09:50:09 the plaintiffs expert witnesses in this
09:50:15 litigation; is that correct?
09:50:15 A. To the extent I'm qualified,
09:50:16 yes.
09:50:18 Q. Including opinions expressed
09:50:21 as to cigarette design and the
09:50:23 manufacturing process?

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09:50:25 A. Yes.
09:50:25 Q. Manufacturing patents?
09:50:29 A. To the extent I'm familiar
09:50:31 with them, yes.
09:50:32 Q. Process specifications?
09:50:32 A. Yes.
09:50:34 Q. Process controls?
09:50:34 A. Yes.
09:50:35 Q. And machinery in the
09:50:37 manufacturing process?
09:50:38 A. Yes.
09:50:48 Q. And you're basing your
09:50:48 opinions offered in this case as an
09:50:52 expert witness on your education,
09:50:52 training and experience?
09:51:05 A. Yes.
09:51:05 Q. And you have stated that you
09:51:08 plan to rely on your review of
09:51:08 pertinent scientific and technical
09:51:07 literature and your own company's
09:51:11 research documents; is that correct?
09:51:12 A. Yes, it is.
09:51:14 Q. And evidence and testimony
09:51:16 that comes forth in this case, you are
09:51:19 prepared to offer opinions on that?

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09:51:22 A. To the extent I'm qualified,
09:51:25 yes.
09:51:28 Q. I want to ask you some
09:51:29 questions about those areas that you
09:51:32 have just identified.
09:51:37 What is a tobacco by-product?
09:51:41 A. Well, when we or anyone
09:51:46 purchases tobacco in a tobacco market,
09:51:50 you are buying the whole leaf which
09:51:53 includes a leafy part of the leaf
09:51:57 itself, and it also contains some
09:52:02 stemming material, sometimes called the
09:52:07 midrib, sometimes called the veins.
09:52:07 And in the course of
09:52:08 processing tobacco, the leafy portion
09:52:11 is removed from the stem and that is
09:52:14 the primary component of a cigarette
09:52:18 blend. However, during the course of
09:52:21 removal, there is some damage to the
09:52:24 leaf. It's a friable agricultural
09:52:27 product so some very small pieces of
09:52:30 tobacco are generated. And there is
09:52:33 also some stem that is separated and
09:52:40 become by-products.
09:52:40 Generally speaking, when I'm

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09:52:41	1	referring to tobacco by-products, I'm
09:52:43	2	talking about the stem material that
09:52:43	3	was part of the leaf that's removed and
09:52:46	4	the small damaged pieces of tobacco
09:52:48	5	which result from the mechanical
09:52:50	6	separation of the leafy portion from
09:52:55	7	the stem.
09:53:01	8	Q. Is the stemming and the
09:53:03	9	removal of the leaf from the stem; is
09:53:07	10	that done manually, or is it done as a
09:53:11	11	part of the manufacturing process and
09:53:13	12	through the equipment that you use?
09:53:20	13	A. Actually, we use contractors
09:53:20	14	to stem tobacco. Primarily in this
09:53:21	15	country its Universal Leaf Tobacco, but
09:53:28	16	there are others as well.
09:53:30	17	It is a mechanical trashing
09:53:32	18	process where whole leaf is introduced
09:53:33	19	into a machine which pulls the leafy
09:53:33	20	portion of tobacco away from the stem,
09:53:38	21	cleans the small pieces of tobacco from
09:53:41	22	the stem, and then separates into two
09:53:44	23	or three different streams -- the large
09:53:48	24	leafy pieces, the stem, and the small
09:53:51	25	tobacco pieces that are damaged as a

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result of the mechanical trashing operations.

Q. In the history -- and I'm not talking about history for the hundreds of years of tobacco growing and manufacture or rolling of cigarettes when I ask this question, just so that I'm clear -- but in the current history of modern tobacco manufacturing, when were by-products first recaptured?

When did you develop systems or procedures to begin to recapture those by-products, or the small products, as you call them?

MR. MUEHLBERGER: Object to the form.

A. I can't give you a precise answer for the industry. To the best of my recollection, Philip Morris began to look for ways to reuse those by-products in the mid-'50s.

BY MR. HOPPER:

Q. Do you know what motivated that decision?

A. Sure. It's primarily

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09:55:00 economic. I mean, tobacco is a very
09:55:03 expensive commodity, a very expensive
09:55:07 raw material. And it contains, as
09:55:12 purchased, about twenty-five percent by
09:55:12 weight, stem.
09:55:13 Plus, there is a three or four
09:55:13 percent loss due to damage in
09:55:17 processing.
09:55:18 So, you might say that of the
09:55:19 whole leaf that the company buys,
09:55:22 without having available reconstituted
09:55:25 tobacco processes, the yield would be
09:55:28 approximately 25, 26, 27 percent less.
09:55:34 Therefore, you would have to
09:55:37 buy a lot more pounds of tobacco to
09:55:39 make the same number of cigarettes.
09:55:42 Q. If the small product wasn't
09:55:44 reconstituted, what would you do with
09:55:44 it?
09:55:51 MR. MUEHLBERGER: Object to
09:55:51 the form.
09:55:52 A. You would have to dispose of
09:55:53 it in some environmental acceptable
09:55:58 way.
09:55:58 BY MR. HOPPER:

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09:55:59 Q. Has Philip Morris done that in
09:56:00 the past?
09:56:02 A. Presumably before they began
09:56:04 using reconstituted tobacco processes,
09:56:06 but it predates me and I don't have any
09:56:09 real experience with it.
09:56:14 Q. Are you able to recapture all
09:56:15 of the by-product that is damaged or
09:56:18 that comes off of the process involved
09:56:20 in the stemming and leaf separation you
09:56:24 described?
09:56:25 A. We try to capture all of it.
09:56:27 Q. Is there a percentage you can
09:56:30 identify for the Jury that you don't
09:56:32 capture?
09:56:39 A. Well, sure.
09:56:39 When you purchase tobacco,
09:56:39 remember most of this leaf is grown in
09:56:41 sandy soil in North Carolina and
09:56:45 Georgia and Florida. So, the rain
09:56:47 hitting the earth splatters some sand
09:56:52 on the tobacco. So, a lot of what you
09:56:55 buy is sand, for example. Obviously,
09:56:58 we don't recover the sand, we try to
09:57:02 remove the sand. When you do that, you

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09:57:06 you lose some tobacco yield as well.

09:57:08 So, my guess is that probably

09:57:12 five or six percent of the purchase

09:57:13 weight is not recovered.

09:57:17 But that is just an estimate.

09:57:19 It's been a long time since I have

09:57:21 looked at the numbers.

09:57:23 Q. Can you tell the Jury what is

09:57:26 reconstituted tobacco?

09:57:28 A. Sure.

09:57:29 Philip Morris uses two

09:57:32 processes to reconstitute tobacco; that

09:57:36 is to take the stem and the small

09:57:39 tobacco pieces that I mentioned a few

09:57:41 minutes ago and convert them into

09:57:45 usable tobacco.

09:57:51 One process is called BL, or

09:57:54 blended leaf, and it's a process by

09:57:58 which tobacco is ground to a very fine

09:57:59 consistency, mixed with certain

09:58:04 chemicals, cast onto a stainless steel

09:58:09 belt, dried, removed from the belt, cut

09:58:11 into small pieces, thereby enabling it

09:58:13 to be used as a cigarette filler blend

component.

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09:58:13 The other process uses the
09:58:27 same starting materials but uses a
09:58:27 paper-making process. It's called the
09:58:27 RL process.
09:58:27 In this process, tobacco stem
09:58:27 and small pieces are mixed with quite a
09:58:30 lot of water. The tobacco solubles,
09:58:34 that part of the plant that is water
09:58:37 soluble, is transferred into the water
09:58:40 or into the liquid. The liquid is
09:58:43 separated from the fibrous material,
09:58:50 which is mostly cellulose. A sheet of
09:58:56 paper is made with the cellulose. A
09:58:52 lot of the water that was added to
09:58:54 remove the solubles is removed and the
09:58:57 solubles are reapplied to the sheet
09:59:00 after a paper machine.
09:59:02 And, therefore, the fiber
09:59:04 portion and the soluble portion are
09:59:29 recombined into a tobacco sheet. It is
09:59:29 cut into fairly small pieces and used
09:59:29 as a cigarette blend component as well.
09:59:31 Q. And the BL process, is that
09:59:34 described as, or does BL represent
09:59:39 blended-leaf process?

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09:59:39 A. Yes.
09:59:40 Q. And RL would be reconstituted
09:59:47 or refined leaf; is that another term?
09:59:47 A. I have heard that term used as
09:59:48 well, yes.
09:59:49 Q. And you referred to the
09:59:51 solubles, is that also the same thing
09:59:57 as the liquor?
09:59:57 A. Yes.
09:59:57 Q. All solubles are considered
09:59:58 the liquor content?
10:00:03 A. When they are in solutions
10:00:04 with water, they are referred to as the
10:00:08 liquor strain. That's a paper-making
10:00:11 term.
10:00:12 Q. And what is RBW and GBW?
10:00:12 A. Well, RBW stands for rich
10:00:17 brown water. I don't know what GBW is.
10:00:19 I have not heard that term before.
10:00:33 Q. What constitutes the liquor
10:00:33 content of the RL process?
10:00:36 MR. MUEHLBERGER: Object to
10:00:36 the form.
10:00:37 A. Can you be a little more
10:00:40 specific? I'm not sure I --

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10:00:40 BY MR. HOPPER:
10:00:43 Q. Well, it's a soluble, correct?
10:00:43 A. Yes.
10:00:43 Q. And what chemicals or compounds make up the soluble content or portion?
10:00:47 A. I don't know.
10:00:48 It's really everything that is in tobacco that is water soluble, and there are probably thousands of compounds in that water soluble component of the tobacco plant.
10:01:03 Q. Are the alkaloids a part of that soluble process?
10:01:06 A. Yes.
10:01:07 Q. Or content, excuse me.
10:01:08 A. Yes, they are.
10:01:08 Q. Is the soluble content primarily alkaloids?
10:01:11 A. No.
10:01:13 Q. Can you identify for me what would be the primary compounds or components? Is there anything that dominates over that soluble over anything else?
10:01:14
10:01:16
10:01:18
10:01:24
10:01:27

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10:01:28	1	A. I can't really, there are a
10:01:38	2	lot of sugars, there are some starches,
10:01:38	3	there are some alkaloids there. There
10:01:42	4	are just thousands of different
10:01:43	5	chemical compounds and I have never
10:01:47	6	really seen a rank ordering of them by
10:01:50	7	concentration.
10:02:01	8	Q. How is the -- when you
10:02:04	9	identified a moment ago that the
10:02:07	10	solubles are separated from the fibers
10:02:13	11	or the cellulose, how are those
10:02:16	12	solubles separated through the
10:02:19	13	manufacturing process?
10:02:19	14	A. Well, the first thing that
10:02:22	15	happens is the stem and small tobacco
10:02:31	16	pieces are conveyed into a very long
10:02:33	17	stainless steel tank. It contains a
10:02:36	18	very large agitator. The tank is
10:02:40	19	filled with water and agitated sort of
10:02:44	20	like a washing machine, for a
10:02:47	21	considerable period of time until the
10:02:47	22	solubles have had a chance to separate
10:02:50	23	from the cellulose.
10:02:50	24	And after some period of time,
10:02:52	25	that tank is discharged and the tobacco

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10:02:58 solubles and the cellulose are pumped
10:03:02 through a device called a Size Press,
10:03:06 which, I suppose you could think of it
10:03:08 as an old-time washing machine ringer
10:03:08 where you have two counter-rotating
10:03:13 rolls. The liquid in the cellulose
10:03:17 portion goes through the nip of the
10:03:19 roll and the solubles are squeezed away
10:03:24 from the fiber. So you have sort of a
10:03:28 crude separation now between the liquid
10:03:32 stream and a fiber stream.
10:03:36 Q. In that process, I can follow
10:03:38 your description as to the solubles
10:03:41 being pumped away. But where do the
10:03:46 fibers of the cellulose remain or go at
10:03:50 that point?
10:03:53 A. Okay. Following the fiber
10:03:58 stream then, it's cleaned up some more
10:03:58 because there is still some residual
10:04:01 solubles there. So it goes through
10:04:04 several stages of rewetting and
10:04:10 separation, just in order to get as
10:04:10 pure a fiber stream as possible.
10:04:14 After that purification
10:04:15 process, you go through a series of

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10:04:18 devices called refiners, exactly the
10:04:23 same equipment that is in a papermill.
10:04:29 And there are simply counter-rotating
10:04:30 discs that are very close together so
10:04:30 that any residual bundles of fibers are
10:04:35 separated into individual fibers. And
10:04:38 after the refining step, the fiber is
10:04:42 pumped to a large chest called a
10:04:45 machine chest. A lot of water is
10:04:48 added. Now you have a very dilute
10:04:51 solution of cellulose fibers and water.
10:04:54 Those fibers and water are then further
10:04:57 diluted, so the concentration of fiber
10:05:00 is about half-a-percent to one percent
10:05:03 fiber and the rest water. They are
10:05:06 applied to a moving wire, or so-called
10:05:10 fordiner on a paper machine. A sheet
10:05:13 of paper is formed. It is removed from
10:05:14 the moving screen by a transfer felt,
10:05:18 dried on a large drum dryer called a
10:05:22 Yankee Dryer. Further dried on some
10:05:26 Can Dryers.
10:05:28 And at that point, you have a,
10:05:35 what we call a base web of tobacco,
10:05:35 which is primarily a cellulose sheet,

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10:05:35 very much like a sheet of paper.
10:05:35 Q. No solubles though, at that
10:05:37 point?
10:05:37 A. Very, very little solubles.
10:05:40 Q. I understood you to say there
10:05:42 was some added back in before you got
10:05:45 to the refinement process, but for the
10:05:48 most part it's fiber?
10:05:49 A. Yes. The objective is to have
10:05:51 no solubles there.
10:05:52 Q. Why it is that in the
10:06:01 manufacturing process of tobacco that
10:06:05 you go through this separation process?
10:06:05 A. You can't -- the solubles are,
10:06:08 first of all, gummy and sticky. You
10:06:09 can't make a sheet of paper with a lot
10:06:12 of tobacco solubles and fiber. You
10:06:15 won't get good sheet formation. And if
10:06:18 you think about how a paper machine
10:06:22 works, it's a screen, it's a moving
10:06:23 screen, just like a screen on your back
10:06:27 door. And what happens is, the fiber
10:06:30 is distributed on that screen and the
10:06:35 water drains through the screen. So,
10:06:35 if the fibers were part of that, they

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10:06:35 would be lost through the screen, then
10:06:36 you wouldn't have the taste of the
10:06:39 tobacco there because the taste is
10:06:41 really in the solubles and not in the
10:06:44 fiber stream.
10:06:46 Q. Wait a second. You lost me
10:06:46 there.
10:06:47 You have, prior to the final
10:06:51 web sheet of the cellulose or the
10:06:54 fibers moving through the process, and
10:06:57 you have purged most of the solubles
10:07:00 out, correct?
10:07:01 A. Yes.
10:07:01 MR. HOPPER: Can you go back
10:07:17 and re-read his answer again, please.
10:07:17 (Reporter read: And if you
10:07:18 think about how a paper machine works,
10:07:22 it's a screen, it's a moving screen,
10:07:27 just like a screen on your backdoor.
10:07:27 And what happens is, the fiber is
10:07:30 diluted on that screen and the water
10:07:30 drains through the screen --)
10:07:30 MR. HOPPER: All right. Stop
10:07:33 right there.
10:07:33 BY MR. HOPPER:

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10:07:33 Q. How is it that the fiber is
10:07:35 diluted through the screen?
10:07:35 A. I probably misspoke.
10:07:35 Q. Yes, I thought --
10:07:41 A. I probably misspoke. What
10:07:41 happens -- let me back up.
10:07:41 The machine chest dilutes the
10:07:42 solution of fiber in water, but before
10:07:47 the sheet of paper can be made, it's
10:07:50 further diluted.
10:07:57 So now, you have a solution
10:08:02 which is, let's say ninety-nine percent
10:08:06 water and one percent fiber. It's
10:08:02 called, "furnish," at this point.
10:08:04 That furnish is distributed
10:08:08 across a moving wire, and as you
10:08:10 deposit this material on a wire, the
10:08:14 fibers stays on a screen, the screen
10:08:15 holds it and the water drains through
10:08:16 the screen.
10:08:16 So, you separate most of the
10:08:19 water that you added on the wire or on
10:08:24 the fordiner portion of the machine.
10:08:25 You get a certain amount of
10:08:27 drying as the screen travels forward.

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10:08:32 There are vacuum boxes to help remove
10:08:32 the water. And you remove as much
10:08:36 water as you can on the wire and then
10:08:39 you transfer the sheet of mostly paper
10:08:43 with a felt, transfer felt, that
10:08:45 carries it from the wire to a large
10:08:47 heated drum called a Yankee Dryer, that
10:08:49 further dries.
10:08:50 After the Yankee Dryer, the
10:08:53 sheet is dry enough to sustain its own
10:08:[illegible] weight, so you can peel it off the
10:08:[illegible] dryer. It's still fairly damp. It
10:09:00 goes through a series of smaller dryers
10:09:00 called Can Dryers.
10:09:[illegible] And then you end up with a,
10:09:[illegible] sort of a finished sheet of paper. Not
10:09:07 a finished sheet of tobacco, because
10:09:[illegible] the solubles have to be reapplied.
10:09:11 Q. Do you consider that finished
10:09:14 sheet tobacco at that point?
10:09:17 A. No.
10:09:17 Q. What is it?
10:09:18 A. It's a cellulose sheet, very
10:09:21 much like a brown paper bag. Because
10:09:21 the fibers -- excuse me -- the solubles

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10:09:26 that give the sheet its character and
10:09:29 its taste have been separated.
10:09:32 Q. If you didn't -- and I
10:09:35 presume as I understand this process,
10:09:38 you do add the solubles back, correct?
10:09:42 A. That's right.
10:09:42 Q. If you didn't, what would you
10:09:45 have or do with the sheet you have at
10:09:47 that point?
10:09:49 MR. MUEHLBERGER: Object to
10:09:49 the form.
10:09:49 A. I guess it would be a
10:09:51 do-nothing process. You would have to
10:09:54 throw it away because you wouldn't want
10:09:57 to smoke it.
10:09:59 BY MR. HOPPER:
10:09:59 Q. Why is that?
10:10:02 A. It tastes bad. It tastes like
10:10:02 smoking a piece of paper.
10:10:02 Q. If I just rolled up a brown
10:10:04 paper bag from the grocery store and
10:10:05 lit it and tried to smoke it at that
10:10:05 point, is that what you're saying it
10:10:05 would taste like?
10:10:11 MR. MUEHLBERGER: Object to the

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10:10:11 form.
10:10:11 A. Well, similar.
10:10:11 BY MR. HOPPER:
10:10:17 Q. So, then, it's your testimony
10:10:19 that you add the solubles back into
10:10:21 that process, that is where the tobacco
10:10:24 gets its flavor?
10:10:26 A. Yes.
10:10:27 Q. And why is that?
10:10:28 A. Because the favorable
10:10:30 components of the tobacco are water
10:10:37 soluble and now that you have been able
10:10:39 to reconstitute the stem into a sheet
10:10:40 form and reapply the solubles, you've
10:10:42 essentially recreated a piece of
10:11:00 tobacco. But it's in a form that is
10:11:00 usable in a cigarette as opposed to a
10:11:00 stem or a small piece of tobacco.
10:11:00 Q. Forgive me, because I'm trying
10:11:00 to understand this.
10:11:02 There is a little bit of a
10:11:02 disconnect for me in why, if the
10:11:06 solubles are so critical to the
10:11:08 finished product, why do you need to
10:11:11 separate them out and then put them

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10:11:13	1	back it? It almost seems dis-economic
10:11:21	2	to do that in a way. Can you explain
10:11:21	3	that to me --
10:11:22	4	MR. MUEHLBERGER: Object to
10:11:24	5	the form.
10:11:25	6	A. Sure.
10:11:25	7	MR. HOPPER: Could you cut me
10:11:25	8	a little bit of slack, Jim? I'm
10:11:26	9	trying to just truly understand
10:11:26	10	this.
10:11:26	11	A. Sure. Remember I described
10:11:27	12	the very dilute fiber solution and the
10:11:27	13	screen.
10:11:27	14	BY MR. HOPPER:
10:11:34	15	Q. Right.
10:11:34	16	A. If the solubles were part of
10:11:34	17	that, if they had not been separated, a
10:11:40	18	couple of things would happen.
10:11:40	19	First of all, the viscosity
10:11:43	20	would be very high, and you wouldn't
10:11:43	21	get good drainage on the wire. You'd
10:11:46	22	plug the wire. And you wouldn't really
10:11:47	23	be able to form a sheet of paper.
10:11:49	24	The other reason is, remember
10:11:51	25	that the liquid components, or the

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10:11:54 water, or the solubles, if we left them
10:11:57 there, would drain through the fiber
10:12:00 and through the screen and they
10:12:02 wouldn't remain with the sheet.
10:12:07 Q. Well, that is what happens
10:12:09 anyway, isn't it, when you are purging
10:12:10 out the solubles?
10:12:11 A. Except that we collect those
10:12:16 solubles. We remove a lot of the water
10:12:20 that was added in order to make them
10:12:23 water soluble, and then we reapply
10:12:26 those solubles to the finish sheet
10:12:30 after the paper sheet is formed, or
10:12:34 after the base web is formed.
10:12:38 Q. And how do you do that?
10:12:41 A. It's done in a device or a
10:12:47 machine called a Size Press.
10:12:48 Again, very similar to
10:12:49 paper-making technology.
10:12:45 The water that was added is
10:12:47 mostly removed in evaporators so the
10:12:49 solubles are now in balance with what
10:12:53 the sheet of paper can absorb.
10:12:55 The paper goes, the dried
10:12:57 paper goes through, again, a set of

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10:12:58 rolls, papers through the middle of
10:13:00 them. And liquid, the liquid soluble
10:13:07 stream is pumped into the nip and as
10:13:11 the paper goes through the nip, it just
10:13:11 reabsorbs like a sponge, the solubles.
10:13:22 And then the sheet is dried to
10:13:22 about twelve percent moisture, cut up
10:13:22 into small pieces, packed out and used
10:13:24 as a tobacco blend component in our
10:13:43 cigarette packers.
10:13:43 Q. And is that process that you
10:13:46 just described of separating out the
10:13:49 fibers from the solubles, is that the
10:13:52 reconstituted tobacco process?
10:13:57 A. Well, both processes are
10:13:58 generically referred to as
10:14:02 reconstituted tobacco process. This is
10:14:07 called the RL process.
10:14:16 Q. When you separate out the
10:14:18 solubles, is that referred to as an
10:14:23 influent process, when you collect it?
10:14:27 MR. MUEHLBERGER: Object to
10:14:28 the form.
10:14:28 A. Not to my knowledge.
10:14:28 BY MR. HOPPER:

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10:14:32 Q. As compared to effluent?
10:14:34 You're not pumping it and draining it
10:14:37 away as waste, correct?
10:14:42 A. No. It's not. It's recovered
10:14:42 and later reused.
10:14:43 Q. And where do you recover it?
10:14:45 Where do you hold it, or contain it?
10:14:48 A. In tanks.
10:14:49 After it's separated, it's
10:14:52 held in tanks. It goes through a
10:14:59 couple of devices to remove any
10:14:59 residual fiber that may be in it,
10:15:04 remove any sand that may be in it. And
10:15:04 then it's pumped to large evaporators,
10:15:04 the same type of device that are used
10:15:09 to concentrate orange juice, because we
10:15:09 have added a lot of water and we have
10:15:12 to remove that before we can apply it
10:15:14 back to the sheet.
10:15:14 So, those solubles are
10:15:18 concentrated in these evaporators and
10:15:23 then reapplied to the sheet in the
10:15:25 right proportion in a Size Press.
10:15:25 Q. And how do you reapply it to
10:15:33 the sheet?

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10:15:33 MR. MUEHLBERGER: Object to the form.

10:15:33 A. In a Size Press.

10:15:35 It was, again, as I described, two counter-rotating rolls, a sheet of paper goes between them, the liquid is pumped into the nip and the sheet just absorbs the solubles like a sponge.

10:15:41 BY MR. HOPPER:

10:15:46 Q. Are there any steps in that manufacturing process that you do or that you treat the solubles when it's separated from the fibers in that stage?

10:15:59 A. Yes.

10:16:01 Actually, there are several. I described a couple of them.

10:16:06 There's some mechanical separations to remove fiber and sand.

10:16:11 We also go through a process to remove as much of the potassium nitrate, which gets into the plant from fertilizer, as we can in a process called denitration or crystallization.

10:16:27 We also add -- after the

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10:16:29 evaporation step -- we also add some
10:16:34 flavors before the solubles are
10:16:37 reapplied to the sheet.
10:16:39 So, I guess in summary, we
10:16:41 have removed some additional fiber and
10:16:45 sand, we removed potassium nitrate and
10:16:45 we have added flavors before reapplying
10:16:48 the solubles to the sheet.
10:16:51 Q. Do you know approximately how
10:16:53 much nitrate is left in that soluble
10:16:58 after you go through the dinitrate
10:17:05 process, denitration process?
10:17:05 A. Yeah, it depends on the crop
10:17:05 year.
10:17:07 Certain years, because of the
10:17:09 growing conditions, contain more or
10:17:13 less potassium nitrate. It's the way
10:17:18 the plant takes it up. But I would
10:17:21 say, sort of an over the years average,
10:17:21 we were removing something over sixty
10:17:25 percent of the potassium nitrate that
10:17:32 is there.
10:17:32 If you had a dry crop year,
10:17:32 for example, where the stems
10:17:34 concentrated more potassium nitrate,

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10:17:34 you would remove more, maybe 65 or 70 percent.

10:17:37 Q. On that issue, it's my understanding that most tobacco farmers are still heavily dependent upon nitrogen fertilizers. Is that your knowledge and understanding?

10:17:49 A. Oh, I think the plant needs nitrogen to grow. So, that is true, yes.

10:17:53 Q. Have you ever -- has your company ever worked with the farmers to set any standards as to the level of nitrogen that they may use?

10:18:01 A. Well, the land grant universities in the U.S. generally work with the farm community to establish fertilization rates.

Our Leaf Department is, you know, very active and collaborative with the land grant universities and they play an advisory role. But I'm not sure exactly, you know, to what level their involvement is.

10:18:39 Q. What is WEL?

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10:18:42 A. WEL stands for weak extract
10:18:46 liquor.
10:18:46 Q. And can you describe what that
10:18:48 is?
10:18:49 A. Sure. I'll take you back to
10:18:53 the pulper, where water is added to dry
10:18:56 tobacco to remove the solubles. And
10:19:01 then we go through a series of presses
10:19:05 to separate the fiber from the liquid.
10:19:09 But it's a three-stage countercurrent
10:19:13 extraction operation or press
10:19:14 operation.
10:19:14 And some fresh water is used
10:19:17 in the last stage to remove as many of
10:19:21 the solubles as you can. And the
10:19:25 liquor stream that results from the
10:19:28 third stage of separation is called
10:19:31 W-E-L. It just contains a very dilute
10:19:34 level of solubles.
10:19:38 Q. And is that different or
10:19:41 distinctively different from SEL?
10:19:44 A. Well, SEL is the more
10:19:46 concentrated liquor from the first
10:19:51 stage of pressing.
10:19:53 Q. Is that, "strong extract

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10:19:56 liquor"?

10:19:56 A. Yes.

10:20:01 Now, all of these streams are
10:20:03 eventually mixed and that water is
10:20:11 removed. So, they're just there for
10:20:11 processing reasons.

10:20:12 Q. Why do you draw a distinction
10:20:18 between the two? What is the point?

10:20:18 A. Again, they are old
10:20:25 paper-making terms. This process is
10:20:25 copied from traditional paper-making
10:20:26 and they do the same thing. And they
10:20:27 just classify the liquor streams based
10:20:29 on the concentrations of solubles, from
10:20:32 weak to strong.

10:20:35 Actually, there are three.
10:20:36 Weak extract liquor, strong extract
10:20:38 liquor and CEL, which is concentrated
10:20:41 extract liquor.

10:20:42 So, it just depends on the
10:20:45 levels of solubles in that particular
10:20:48 stream.

10:20:54 Q. Of WEL, SEL and CEL, which is
10:20:59 the concentrated extract liquor, is
10:21:05 there any separation out of the

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10:21:07 compounds distinct to those three
10:21:10 liquors? Or are they found in all of
10:21:16 those liquors?
10:21:18 A. They are found in all of them.
10:21:28 Q. You don't have alkaloids in
10:21:28 SEL but not in WEL or CEL?
10:21:39 A. No.
10:21:[illegible] Q. With respect to the alkaloids
10:21:54 in the solubles, have you ever taken
10:22:03 any steps -- does the company ever
10:22:[illegible] attempt to measure the content of the
10:22:[illegible] solubles?
10:22:[illegible] A. No.
10:22:09 I mean, I have for some
10:22:10 special studies from time-to-time used
10:22:14 alkaloids as a tracer, but it's not a
10:22:17 routine production measurement. So,
10:22:20 the company, on a day-to-day basis of
10:22:21 running its plants doesn't pay any
10:22:27 attention at all to the alkaloid level.
10:22:27 Q. Has there ever been a unit in
10:22:31 Philip Morris called TQAF that ever
10:22:35 measured alkaloid content in solubles?
10:22:45 A. I don't think in solubles.
10:22:45 TQAF was a laboratory called the

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10:22:45 Tobacco Quality Audit Facility, and
10:22:47 their function was to look at the
10:22:49 chemistry of purchased tobaccos.
10:22:57 I think from time-to-time they
10:22:57 also did audits on reconstituted
10:22:59 tobacco in some of our blends as well.
10:23:03 But again, this would have
10:23:06 been a long, after-the-fact audit
10:23:08 measurement.
10:23:12 Q. Is that Tobacco Quality Audit
10:23:12 Facility within Philip Morris? It's
10:23:12 not an outside agency or unit, is it?
10:23:15 A. It was within Philip Morris.
10:23:16 We, several years ago, stopped
10:23:19 doing the leaf analysis.
10:23:21 Q. And why is that?
10:23:22 A. Well, there was a belief, a
10:23:25 dream, I guess, that if you knew enough
10:23:27 about the chemistry of the leaf, that
10:23:32 you could put together leaf blends that
10:23:35 would taste the same without relying on
10:23:39 a lot of humans experts to smoke a lot
10:23:43 of samples. We just never could get
10:23:48 good correlation.
10:23:49 So, it turned out to be an

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10:23:54 expensive process and very little, if
10:23:54 any, value. So, we stopped doing it.
10:23:56 It was a large experiment that occurred
10:23:58 over several years.
10:24:01 Q. From when to when?
10:24:02 A. I don't remember. I'm sorry,
10:24:03 I just don't remember the dates.
10:24:07 Q. Of the alkaloid portion of the
10:24:09 solubles, other than nicotine, what
10:24:13 else would be included among the
10:24:17 alkaloids?
10:24:18 What other chemicals or
10:24:21 compounds would comprise the alkaloids?
10:24:25 A. Well, nicotine is, by far, the
10:24:28 largest alkaloid in tobacco. There are
10:24:32 some other minor alkaloids. Nornicotine
10:24:37 for example.
10:24:37 But they're in very, very
10:24:41 small concentrations.
10:24:41 For the most part, almost all
10:24:42 of the alkaloids in tobacco are
10:24:48 nicotine.
10:24:48 Q. Do you have a figure you might
10:24:48 put on that?
10:24:49 A. Oh, I guess, just to put it in

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10:24:52 perspective for you, probably 95
10:24:56 percent or more.
10:25:00 THE WITNESS: Do you mind if
10:25:01 we take a short break?
10:25:05 MR. HOPPER: No. Would be
10:25:06 happy to.
10:25:06 THE WITNESS: Thank you.
10:25:07 VIDEOGRAPHER: Going off the
10:25:09 record at 10:25 A.M.
10:25:09 (Off record.)
10:36 VIDEOGRAPHER: Returning back
10:36 on the record at 10:36 A.M.
10:36:14 BY MR. HOPPER:
10:36:20 Q. Mr. Burnley, is it at this
10:36 stage of the solubles, before you
10:36:36 recombine them with the fibers, that
10:36:41 the humectants and preservatives and
10:36:41 flavoring is added?
10:36:41 A. Yes.
10:36:41 Q. And can you describe that
10:36:41 process and procedure?
10:36:41 A. It's pretty simple.
10:36:41 The solubles are pumped to a
10:36:43 tank where the other additives are
10:36:47 added. And then the mixture is pumped

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10:36:51 out of the tank to the Size Press as I
10:36:57 described it a little while ago, for
10:36:58 reapplication to the sheet.
10:36:59 Q. Why do you add flavors to the
10:37:05 solubles?
10:37:06 A. To flavor the sheet. To make
10:37:15 it subjectively more acceptable.
10:37:15 Q. What do you mean when you say,
10:37:15 "subjectively more acceptable"?
10:37:19 A. Make it taste better.
10:37:21 Q. To an individual who might
10:37:23 smoke the tobacco?
10:37:25 A. Yes.
10:37:26 Q. Isn't it true that you also
10:37:29 add those flavors to make the product
10:37:33 more inhalable?
10:37:41 MR. MUEHLBERGER: Object to
10:37:41 the form.
10:37:41 MR. HOPPER: What is your
10:37:41 objection?
10:37:41 MR. MUEHLBERGER: I don't know
10:37:41 what you mean by inhalable. At
10:37:41 what point in time?
10:37:41 MR. HOPPER: At what point in
10:37:41 time? At any point in time someone

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10:37:41 would inhale.
10:37:41 BY MR. HOPPER:
10:37:41 Q. Isn't it true that you add
10:37:43 those flavors to make the product more
10:37:50 inhalable?
10:37:50 A. I'm not sure I understand what
10:37:50 you mean by more inhalable.
10:37:54 Certainly, when you add
10:37:56 flavors, it make the smoking experience
10:37:59 more pleasant, the smoke taste better.
10:38:00 Q. Less harsh?
10:38:00 A. Could be, yes.
10:38:02 Q. Less caustic on the back of
10:38:06 the throat?
10:38:07 A. Presumably, that's possible.
10:38:10 Q. Mechanically easier to draw
10:38:12 down the throat and into the lungs?
10:38:16 MR. MUEHLBERGER: Object to
10:38:17 the form.
10:38:17 A. Mechanically easier, I kind of
10:38:24 doubt that.
10:38:24 BY MR. HOPPER:
10:38:24 Q. And what do the humectants do?
10:38:25 A. Well, tobacco dries out very
10:38:28 readily. And remember, this tobacco is

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10:38:32 going to undergo a lot more processing,
10:38:36 it's going to be combined with other
10:38:39 tobaccos, other reconstituted tobaccos,
10:38:41 cut and made into a cigarette.
10:38:45 The humectants tend to retain,
10:38:48 help retain moisture to make the
10:38:48 tobacco soft and pliable so it can be
10:38:49 formed into a cigarette without turning
10:38:51 to dust.
10:38:52 Q. And you add preservatives for
10:38:56 what purpose?
10:38:56 A. To make sure there is no
10:38:59 microbial action taking place that
10:39:04 could have the effect of spoiling the
10:39:08 RL.
10:39:09 Q. There are microbes that come
10:39:15 in from the raw feed stock into the
10:39:16 manufacturing process that are retained
10:39:19 in the tobacco and you add the
10:39:19 preservatives to destroy those
10:39:24 microbes?
10:39:24 A. That is approximately correct,
10:39:27 yes.
10:39:28 Q. There is a term that I believe
10:39:29 your company uses called POL. Can you

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10:39:29 tell me what stands for?
10:39:32 A. Yeah, it stands for public
10:39:34 opinion laboratory. And it's just our
10:39:36 way to evaluate our product using a
10:39:41 panel of consumers.
10:39:43 So, if we make a change to the
10:39:46 product and we want to evaluate how the
10:39:50 consumer may react to that change,
10:39:53 accept that change, we send product to
10:39:57 the consumer and ask them to fill out a
10:39:[illegible] ballot about the product.
10:39:[illegible] Q. Do you invite consumers to the
10:40:01 company or to agents that the company
10:40:[illegible] may employ to run focus groups?
10:40:[illegible] MR. MUEHLBERGER: Object to
10:40:11 the form.
10:40:11 A. We do use focus groups as
10:40:13 well, yes.
10:40:13 BY MR. HOPPER:
10:40:14 Q. Would that be considered a
10:40:15 part of this public opinion laboratory?
10:40:19 A. No. Public opinion laboratory
10:40:22 is used for external sensory evaluation
10:40:37 of the product.
10:40:37 Q. And what would sensory

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10:40:37 evaluation entail?
10:40:37 A. We ask the consumer really two
10:40:37 questions. How would you compare this
10:40:37 product to your current product in
10:40:39 terms of strength and the like, is it
10:40:39 stronger, or less strong, do you like
10:40:47 it more, do you like it less.
10:40:47 Q. Strong in terms of what
10:40:48 variable?
10:40:49 A. However the consumer
10:40:53 interprets it.
10:40:53 Q. And what is your feedback,
10:40:54 then? What does the consumer typically
10:40:58 interpret as being strong or not
10:40:58 strong?
10:40:58 MR. MUEHLBERGER: Object to
10:40:59 the form.
10:41:00 A. I don't know. I mean, this is
10:41:04 a subjective evaluation, the customer
10:41:04 is saying, compared to my current
10:41:08 product, I think this product is
10:41:10 stronger or milder and I like it more
10:41:17 or I like it less.
10:41:17 BY MR. HOPPER:
10:41:17 Q. But what would that consumer

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10:41:19 be telling you that they're evaluating
10:41:20 to derive at a label of stronger or
10:41:20 weak?
10:41:26 A. They don't.
10:41:26 Q. You have never heard that term
10:41:27 identified or defined?
10:41:30 A. No.
10:41:31 Q. Is it possible it's defined in
10:41:34 terms of the taste.
10:41:36 MR. MUEHLBERGER: Object to
10:41:38 the form.
10:41:38 A. It's however the customer
10:41:39 defines it.
10:41:40 I mean, there aren't any
10:41:42 instructions on what is more or less
10:41:46 strong or what is better or more.
10:41:48 Q. What does the company like to
10:41:50 get in the way of feedback on a range
10:41:54 of variables as to those opinions from
10:41:57 the laboratory?
10:41:58 A. It's typically used to
10:41:59 evaluate changes to a product. So, we
10:42:02 have a history or a data base on how
10:42:05 consumers rate their products.
10:42:07 Let's assume it's Marlboro.

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10:42:26 So, we have a profile for Marlboro, a certain strength rating and a certain liking rating. And we simply --

Q. Excuse me. Liking?

A. Liking rating.

Q. Liking, L-I-K-I-N-G?

A. Right. It's a numerical score.

10:42:26 And we give them a product to smoke that represents a change in something, and we ask them the same two questions. How does it compare with your current product in terms of strength or liking.

And if the differences are large, it tells us that the customer is going to perceive a difference in these products and we had better think pretty hard about making a change.

10:42:57 Q. And flavor certainly is a strong component of that, correct?

10:42:57 A. Sure.

10:42:59 Q. And are such things that you do as a part of this POL process 10:43:00 related to blend component studies?

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10:43:01 A. Sure. I mean, if we changed a
10:43:04 blend component, we would ask the
10:43:08 consumers to evaluate the change and
10:43:12 see how he or she perceived the change.
10:43:14 Q. And what is a blend?
10:43:16 A. A blend is really a recipe of
10:43:23 the individual tobacco types that are
10:43:27 used to make up the cigarette filler.
10:43:30 It could be different grades
10:43:37 of flue-cured, different grades of
10:43:38 Burley, changes to the RL flavor
10:43:39 system, for example, change to a
10:43:42 processing condition in reconstituted
10:43:43 tobacco.
10:43:48 It could be the amount of
10:43:49 oriental tobacco used. It could be the
10:43:49 amount of expanded tobacco or expanded
10:43:53 stem that's used.
10:43:55 It could be any number of
10:43:58 changes that could affect the blend
10:43:59 recipe for the cigarette.
10:44:03 Q. Can the consumer evaluate down
10:44:07 to those differentials between blends?
10:44:11 A. Well, we -- our leaf blenders
generally work with the blend and make

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10:44:14 a modification that they believe the
10:44:17 consumer will not be able to
10:44:21 differentiate.
10:44:21 And this is sort of a final
10:44:22 test. I mean, the leaf blenders do the
10:44:24 work to make the substitution or to
10:44:27 make the change, and this is just a
10:44:30 confirmation with the consumer that,
10:44:3[illegible] it's okay, I can't tell the difference.
10:44:[illegible] Q. Isn't it true that one of the
10:44:[illegible] effects that you like to have feedback
10:44:[illegible] on, though, is the inhalability of your
10:44:[illegible] product?
10:44:[illegible] MR. MUEHLBERGER: Object to
the form.
10:44:[illegible] A. I have never heard any measure
10:44:51 referred to as inhalability.
10:44:[illegible] Q. But under that auspice of
10:44:56 whether it's strong or weak, isn't that
10:44:58 typical of the feedback that you get?
10:45:01 A. I don't think it's typical of
10:45:03 the feedback we get. I suppose if the
10:45:06 smoke were irritating, the consumer
10:45:06 would either say it's stronger or they
10:45:09 don't like the product as much.

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10:45:09 Q. And irritating in what respect
10:45:14 when you use that word?
10:45:14 A. Irritating in the throat.
10:45:26 Throat or mouth or lungs.
10:46:57 MR. HOPPER: Can we go off the
10:46:59 record for a minute. I just want
10:46:59 to take a real quick break.
10:47:02 VIDEOGRAPHER: Going off the
10:47:03 record at 10:46 A.M.
10:47:21 (Off record.)
10:49:04 VIDEOGRAPHER: Going back on
10:49:04 the record at 10:48 A.M.
10:49:04 BY MR. HOPPER:
10:49:04 Q. Mr. Burnley, in the removal of
10:49:04 the solubles, you described for the
10:49:06 Jury a moment ago that nicotine is the
10:49:06 largest alkaloid within all of the
10:49:11 alkaloid content in those solubles as
10:49:11 about ninety-five percent; is that
10:49:13 correct?
10:49:13 A. Yes.
10:49:21 Q. Isn't it true that there have
10:49:24 been times when you have extracted
10:49:26 nicotine -- nicotine alkaloid -- out of
10:49:40 the soluble process?

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10:49:40 A. Not to my knowledge.
10:49:40 Q. Wasn't there a denic program
10:49:40 or a project at one time in the recent
10:49:44 history of Philip Morris?
10:49:45 A. Yes, but that didn't extract
10:49:48 nicotine from the soluble component of
10:49:54 RL as you described.
10:49:54 Q. Describe for me what the
10:49:57 project or process did.
10:49:57 A. Well, it was actually a pretty
10:50:02 interesting process that used
10:50:08 supercrictical carbon dioxide to
10:50:16 extract nicotine from cut tobacco.
10:50:19 Q. And what was the purpose of
10:50:22 that extraction?
10:50:24 A. To enable us to market a
10:50:26 nearly nicotine-free cigarette.
10:50:28 Q. Did you, in fact, market that?
10:50:34 A. We did.
10:50:24 Q. And what product was that?
10:50:26 A. There were several variants.
10:50:28 There were a lot of test-markets.
10:50:33 One was called Merit Denic.
10:50:37 Another trademark was Next. And there
10:50:52 were a couple of others.

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10:50:52 Q. Was there Accord?
10:50:52 A. Accord is a different product.
10:50:52 Q. What is Accord?
10:50:52 A. Accord is an electrically heated cigarette.
10:50:53 Q. Were you involved in that process development and product development?
10:50:57 A. Yes.
10:50:58 Q. Can you tell me what your role was with Accord?
10:51:02 A. Sure. First of all, I was part of the management team that made a lot of the strategic decisions during the course of the development of Accord.
But my direct development was developing the tobacco mat that was used to generate the tobacco aerosol, flavored aerosol --
10:51:21 Q. Excuse me. Tobacco mat?
10:51:21 A. Mat.
10:51:22 Q. Thank you.
10:51:22 A. Which generated -- which was electrically heated and generated
10:51:25

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10:51:27 tobacco aerosol which gives the
10:51:31 consumer the tobacco taste from this
10:51:33 product.
10:51:34 Q. And you test-marketed that
10:51:39 product?
10:51:39 A. Yes.
10:51:40 Q. How far or widely did you
10:51:40 test-market it?
10:51:41 A. It's currently being
10:51:42 test-marketed in a couple of hundreds
10:51:45 stores in Richmond, Virginia. It's
10:51:49 also being test-marketed in quite a few
10:51:52 stores in Japan, in Osaka.
10:51:52 Q. Has your company or other
10:51:57 colleagues of yours made a
10:51:59 determination that Accord is
10:52:00 commercially viable at this time?
10:52:02 A. We believe it is commercially
10:52:06 viable.
10:52:08 Q. How would you compare, other
10:52:10 than the fact that Accord is
10:52:12 electrically heated versus smoked; how
10:52:18 would you compare it to the regular or
10:52:22 typical brands of tobacco that Philip
10:52:24 Morris markets?

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10:52:25 A. From what point of view?
10:52:27 Q. Well, from any of the points
10:52:30 of view that you are involved in with
10:52:34 the process development and/or the
10:52:35 manufacturing process that you just
10:52:38 spent so long a time laboring to define
10:52:40 and describe for me.
10:52:44 A. Well, it's a very, very
10:52:44 different product.
10:52:44 First of all, the tobacco is
10:52:47 prepared on a process that is very
10:52:51 similar to the RL process, where ground
10:52:54 -- or a sheet is made very much like
10:52:59 RL, and then ground tobacco, ground
10:53:03 Marlboro filler, for example, is
10:53:06 applied, is coated on the sheet.
10:53:08 And this mat then is formed
10:53:15 into a ring and there are eight
10:53:18 heaters, electrically operated heaters,
10:53:19 around the ring that fire when someone
10:53:22 puffs on a cigarette to generate a
10:53:26 tobacco aerosol that is roughly the
10:53:29 equivalent of an ultra-low delivery
10:53:34 product.
10:53:34 Q. That is the question I was

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10:53:36 trying to get at.

10:53:37 What is an ultra-low delivery

10:53:37 product?

10:53:43 A. It's a cigarette that would

10:53:44 generally deliver a tar of four or

10:53:51 five, six milligrams.

10:53:52 Q. And how much nicotine would it

10:53:54 deliver?

10:53:56 A. I don't recall precisely. But

10:54:00 it would be pretty much in proportion

10:54:04 with conventional cigarettes. So, the

10:54:06 nicotine delivery is very normal for

10:54:09 that tar delivery.

10:54:12 Q. How do you measure the level

10:54:14 of nicotine in a cigarette, ultra-low

10:54:14 delivery cigarette.

10:54:16 A. Use the FTC tar and nicotine

10:54:20 system.

10:54:20 Q. Do you measure it -- excuse

10:54:24 me. Were you finished?

10:54:25 A. Yes, I'm done.

10:54:25 Q. Do you measure it by PH?

10:54:28 A. I'm sorry. By what?

10:54:29 Q. Do you measure it also by PH?

10:54:32 A. By PH? No.

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10:54:33 Q. What would the PH be of an
10:54:38 ultra-low delivery cigarette?
10:54:38 MR. MUEHLBERGER: Object to
10:54:38 the form.
10:54:41 A. You're going to have to be
10:54:41 more specific. I'm not sure I
10:54:41 understand what you are referring to.
10:54:43 Q. Is there an average PH of
10:54:47 about 5.5 in an ultra-low cigarette?
10:54:47 A. If you're talking about smoke
10:54:53 PH, the smoke PH of our products run
10:54:59 around five, five-one, five-two,
10:55:01 generally speaking.
10:55:03 Q. What are some of the other
10:55:03 distinguishing characteristics between
10:55:08 Accord and an ultra-low?
10:55:08 A. Well, the tar, the composition
10:55:09 of the tar is very different for this
10:55:14 product.
10:55:14 A lot of compounds that the
10:55:16 public health community has targeted as
10:55:20 problematic, being harmful, are a
10:55:26 function of the combustion temperature
10:55:28 during smoking. Combustion -- there is
10:55:34 no combustion in this product. The

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10:55:38 cigarette is heated at a relatively low
10:55:40 level. Therefore, the composition of
10:55:41 the tar is quite different.
10:55:46 A lot of the substances that
10:55:46 public health authorities have targeted
10:55:50 as being problematic are very
10:55:51 dramatically reduced. Sometimes
10:55:58 eliminated altogether.
10:56:01 The cigarette produces no
10:56:01 sidestream because it's puff on demand.
10:56:02 The consumer has no control
10:56:05 over the puff size because the
10:56:08 electronics determine that
10:56:10 automatically.
10:56:11 The product is inherently fire
10:56:15 safe because there is no burning coal,
10:56:17 there is only a heat source when the
10:56:20 consumer decides to take a puff.
10:56:30 So, there are a lot of
10:56:34 differences between this product and
10:56:44 conventional products.
10:56:49 Q. What is the PH of the Accord?
10:56:55 A. I don't know.
10:56:55 Q. In the smoke?
10:56:55 A. I don't know.

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10:56:55 Q. Who would know?

10:56:56 A. Well, I have never really seen
10:56:58 it measured in any of the data sheets
10:57:01 or any of the meetings I have ever been
10:57:05 to.

10:57:05 I guess it I were going to
10:57:07 ask, I would ask Sue Wren.

10:57:09 Q. And who is she?

10:57:10 A. She is in charge of product
10:57:16 development for Accord.

10:57:16 Q. R-E-N-T?

10:57:17 A. No, W-R-E-N.

10:57:28 Q. Is the nicotine content in
10:57:31 Accord more or less than the ultra-low
10:57:34 or conventional cigarette?

10:57:35 A. It's pretty proportional. Per
10:57:39 milligram of tar, it would be right in
10:57:43 the range with conventional cigarettes.

10:57:46 Q. And what would that be?

10:57:49 A. I don't know off the top of my
10:57:48 head. I would have to look at a pack.
10:57:50 I just don't remember.

10:59:08 Q. Is the Accord -- excuse me.
10:59:11 Let me restate that.

10:59:13 Does Philip Morris still

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10:59:17 convene Vision 2000? Are you familiar
10:59:32 with that?
10:59:32 A. No.
10:59:32 Q. That's no longer held or
10:59:32 considered a part of the planning
10:59:32 process in strategic planning in Philip
10:59:35 Morris for new products?
10:59:38 A. I don't ever remember it, to
10:59:40 be honest with you.
10:59:42 Q. P-98?
10:59:44 A. P-98, yes.
10:59:48 Q. And then followed by P-2000 or
10:59:51 Vision 2000?
10:59:51 A. Okay, perhaps. P-98 was
10:59:55 simply a way to look several years down
10:59:59 the road at what changes we wanted to
11:00:03 make to the product and get the right
11:00:06 technical resources focused on making
11:00:06 those changes, so you have a particular
11:00:06 goal to shoot for.
11:00:09 I don't really remember Vision
11:00:12 2000, but it may have been something
11:00:14 similar.
11:00:16 Q. Would Accord have been
11:00:18 conceived as a part of these processes

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11:00:22 or planning processes?

11:00:24 A. No. Actually, Accord predates
11:00:28 both of those.

11:00:30 Q. When was Accord
11:00:31 conceptualized?

11:00:33 A. Again, I'm going to have to
11:00:38 give you an approximate answer. I
11:00:39 would say very late-'80s.

11:00:44 Q. And do you know the motivation
11:00:48 behind the development of Accord?

11:00:51 A. Sure. We initially decided to
11:01:01 market a product that would respond to
11:01:11 a product Reynolds had introduced
11:01:11 called Premier.

11:01:11 We experimented with that for
11:01:12 a while and gave up on the concept
11:01:14 because the product didn't taste like a
11:01:23 cigarette.

11:01:25 However, we knew as a company
11:01:28 that certain chemicals constituents
11:01:27 could be prevented from being formed if
11:01:31 you could lower the combustion
11:01:35 temperature. So the challenge was to
11:01:40 figure out how to lower the combustion
11:01:43 temperature.

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11:01:43 And a number of ideas
11:01:46 surfaced, including electrically heated
11:01:54 cigarettes.
11:01:55 At the time, the battery
11:01:55 technology and metals technology and
11:01:55 miniaturization of electronics had not
11:01:55 evolved to the point where it was
11:01:56 commercially feasible.
11:01:59 But the company began
11:01:59 experimenting with the concept, red-
11:02:04 boarding techniques and car batteries
11:02:04 and very early prototypes to prove the
11:02:04 concept and begin to measure changes in
11:02:13 smoke chemistry.
11:02:14 We also wanted to reduce the
11:02:18 annoyance factor of smoke and a lot of
11:02:19 the odor. So, the idea of
11:02:19 puff-on-demand so the cigarette was not
11:02:23 burning between puffs, or smoldering
11:02:26 between puffs, became an objective as
11:02:30 well.
11:02:30 So over time, as we learned
11:02:32 more and as there were technology
11:02:43 advances in metals and electronics and
11:02:43 battery technology, we began to put

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11:02:43 1 together some commercial prototypes of
11:02:53 2 this product and a couple of years ago
11:02:53 3 introduced them into test-markets.
11:02:53 4 And I will tell you that the
11:02:54 5 first couple of models didn't taste
11:02:54 6 very good. But we made a lot of
11:02:57 7 improvements subjectively to the
11:02:59 8 product. We have gotten the product
11:03:02 9 smaller.
11:03:03 10 You've got to understand that
11:03:07 11 the smoking ritual is very different.
11:03:07 12 It's probably not for everybody and it
11:03:07 13 takes some getting used to.
11:03:09 14 But we have stuck with the
11:03:12 15 product. We are proud of the product.
11:03:16 16 I think the smoke chemistry data is
11:03:19 17 pretty interesting, pretty clean.
11:03:22 18 Q. What do you mean by that?
11:03:25 19 A. I mean that a lot of the
11:03:26 20 compounds that have been targeted by
11:03:32 21 public health authorities as being
11:03:32 22 harmful are significantly reduced.
11:03:34 23 Q. Can you identify some of the
11:03:36 24 those constituents?
11:03:38 25 A. Sure. Certain aldehydes,

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11:03:39 certain polyaromatic hydrocarbons, carbon monoxide, benzo(a)pyrene. There are a whole host of them.

Q. Can you describe for me the Next product that you referred to a moment ago?

A. The denicotinized Next?

Q. Yes.

A. Sure. It was a conventional cigarette made from tobacco that had been supercritically extracted to remove -- I think our target was to remove ninety-seven percent of the nicotine.

Q. And why was that a goal or an objective?

A. Through some focus group testing, we believed that the consumer was interested in a nicotine-free product.

Q. Why?

A. Because they told us they were.

Q. But what was their motivation when they told you that?

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11:05:42 MR. MUEHLBERGER: Object to the form.
11:05:43 A. I don't know.
11:05:43 BY MR. HOPPER:
11:05:43 Q. Did they tell you that because they believed nicotine was addictive?
11:05:45 A. I don't know. I was not really part of the focus group. Never really saw the research.
11:05:48 Q. Did you confer with any of the people in marketing in your position as Vice-President of Process Development in ramping up for the manufacture of this product?
11:05:49 A. Well, we had routine meetings with the entire launch product team, which included people from marketing.
11:06:01 Q. Was any of the -- was any of the feedback received about denicotinizing the Next product related to addiction?
11:06:07 MR. MUEHLBERGER: Object to the form.
11:06:21 A. Not that I recall.
11:06:21 BY MR. HOPPER:

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11:06:21 Q. Was it ever discussed?
11:06:22 A. I don't recall it being
11:06:24 discussed.
11:06:25 Q. You have never been a part of
11:06:27 discussions at Philip Morris with any
11:06:29 of your colleagues where addiction was
11:06:33 discussed?
11:06:34 A. To be honest with you, not
11:06:37 since the litigation began in late '94.
11:06:47 Q. And you're testifying today to
11:06:48 the Jury that in the discussions in
11:06:52 planning and strategic development of
11:06:54 Next, that denicotinizing that product
11:06:59 had nothing to do with either feedback
11:07:04 related to addiction?
11:07:04 MR. MUEHLBERGER: Object to
11:07:06 the form.
11:07:06 A. Not to my knowledge.
11:07:08 I mean, sure, you would have
11:07:09 some watercooler conversations where
11:07:13 people would say, well, it's not going
11:07:16 to sell because it doesn't have
11:07:18 nicotine. But yet you had a lot of
11:07:20 other people say, people smoke for a
11:07:20 lot of reasons and the consumer was

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11:07:20 telling us that they would like to have
11:07:22 a nicotine-free product, so let's try
11:07:22 to provide one.
11:07:22 Q. In their telling you they
11:07:23 would like to have a nicotine-free
11:07:26 product, was the feedback ever based on
11:07:30 the fact that they were concerned about
11:07:31 addiction?
11:07:32 MR. MUEHLBERGER: Objection.
11:07:49 Asked and answered.
11:07:49 A. I don't know.
11:07:49 BY MR. HOPPER:
11:07:49 Q. Who would know?
11:07:49 A. To tell you the truth, I don't
11:07:49 know today who would.
11:07:49 I guess if wanted to track it
11:07:50 down, I would go to the Senior
11:07:54 Vice-President of Marketing and ask her
11:08:14 who was involved at that time.
11:08:14 Q. And what years was Next in the
11:08:14 developmental stages at Philip Morris?
11:08:14 A. 1988 through, probably -- I'm
11:08:16 trying to remember now. 1993, '94.
11:08:22 Q. And were you -- I'm sorry. I
11:08:23 cut you off. I thought you were

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11:08:23 finished. Would you repeat that last
11:08:24 part.
11:08:24 A. I said that was the sort of
11:08:27 development and commercialization time
11:08:30 frame.
11:08:30 Q. Was Next ever marketed as a
11:08:33 product?
11:08:34 A. Yes.
11:08:34 Q. Is it still available on the
11:08:36 market?
11:08:37 A. No.
11:08:37 Q. How long was it marketed?
11:08:39 A. A couple of years.
11:08:41 Q. Was it a marketed as a fully
11:08:41 commercially viable product of Philip
11:08:44 Morris, or was it marketed as a
11:08:47 test-market product?
11:08:47 A. It was in test-market.
11:08:52 Q. Okay, never reached the level
11:08:53 where you fully branded it and marketed
11:08:54 it and commercialized it?
11:08:57 A. Well, it was branded and it
11:08:58 was commercialized. We built a huge
11:08:59 plant. We had a lot of capacity.
11:08:59 But we went into a number of

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different test-markets. I mean, we were still experimenting with flavor systems and, you know, trying to get the product right, get some feedback from the consumer; but it never really took hold.

I mean, there were some initial tries, like a lot of new product introductions but it trickled off to pretty low demand and it was hard to convince retailers to keep it on the shelf because their space is valuable. It was hard to convince wholesalers to carry the product, so eventually we withdrew it.

Q. And how long was it on the market?

A. I'm approximating it, but a couple of years.

Q. And do you know approximately how much Philip Morris invested on gross on that denic plant or facility?

A. I know very well. It was around three hundred million dollars.

Q. Were you involved in the

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11:09:56 design and in the process development
11:09:58 of that plant?
11:09:59 A. I was.
11:10:00 Q. And what was your role and
11:10:01 responsibility?
11:10:02 A. Well, at the time I was
11:10:03 Director of Process and Project
11:10:08 Engineering, so the conceptual design
11:10:09 of the plant was done by my department
11:10:12 and a lot of my engineers managed the
11:10:15 commercial design and construction of
11:10:18 the facility.
11:10:18 Q. Did you believe in the Next
11:10:23 product?
11:10:23 A. I did. I was excited about
11:10:24 it.
11:10:24 Q. Why?
11:10:24 A. It was a great piece of
11:10:25 technology. It worked really well. I
11:10:28 had always believed that there was a
11:10:31 lot more to the smoking experience than
11:10:32 nicotine. I thought it was a niche
11:10:37 market for the company. We had great
11:10:40 technology and I thought it would be a
11:10:44 successful product.

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11:10:44 Q. Was your view contrary to most
11:10:49 of the conventional thinking of your
11:10:52 colleagues at Philip Morris at the
11:10:53 time?
11:10:54 A. I mean, I would say not.
11:10:54 Because, you know, you've got to get a
11:10:54 Board of Directors to approve a huge
11:10:55 investment.
11:10:55 So, I've got to tell you, I
11:10:58 think the company's management believed
11:11:00 the product would be successful or it
11:11:04 wouldn't have spent three hundred
11:11:07 million dollars.
11:11:07 Q. Well, why wasn't it
11:11:07 successful?
11:11:08 A. I don't know. There aren't
11:11:10 very many new cigarette products that
11:11:13 are.
11:11:15 Q. When you ramped up, for lack
11:11:18 of a better way to put it, the denic
11:11:21 facility, how did you go about
11:11:27 extracting the nicotine from the
11:11:32 solubles? Or the alkaloids from the
11:11:32 solubles?
11:11:32 A. Right.

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11:11:32 Q. More appropriately asked.
11:11:32 A. Well, there was a so-called
11:11:34 extraction loop, contained two very
11:11:37 large pressure vessels. Tobacco was
11:11:42 placed in one vessel and stem was
11:11:47 placed in another vessel. And the
11:11:49 purpose of the stem was to absorb the
11:11:58 nicotine that was removed from tobacco.
11:11:58 In high pressure,
11:11:58 supercrictical carbon dioxide was
11:12:00 circulated through that loop, removing
11:12:05 nicotine from tobacco and depositing it
11:12:08 on the stems.
11:12:11 Q. How did you effectively go
11:12:14 about depositing the alkaloids back on
11:12:18 to the stems?
11:12:18 A. Well, the stems are generally
11:12:24 low in nicotine and the mass transfer,
11:12:29 or the driving force, tends to be from
11:12:30 an area of high concentration to an
11:12:33 area of low concentration.
11:12:33 Q. Can I stop you there just so
11:12:33 I'm clear?
11:12:35 A. Sure.
11:12:35 Q. From the leaf to the stem, you

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11:12:38 1 are saying?
11:12:38 2 A. Right.
11:12:39 3 Q. And that is in the raw feed
11:12:45 4 stock coming into the plant, correct,
11:12:45 5 that you are describing it; at that
11:12:46 6 point in time?
11:12:47 7 A. We started with cut tobacco,
11:12:49 8 finished cigarette filler, put it in a
11:12:53 9 large vessel, put the stem in another
11:12:57 10 large vessel and circulate at high
11:12:58 11 pressure CO2 through the loop.
11:13:01 12 It extracted the nicotine from
11:13:04 13 the tobacco and reapplied it or
11:13:05 14 redeposited it on the stem.
11:13:09 15 Q. Was that technology available
11:13:12 16 prior to the conceptualization and
11:13:16 17 development of the Next product?
11:13:18 18 A. Well, we had -- Philip Morris
11:13:21 19 had bought -- purchased General Foods a
11:13:26 20 year or so earlier. And General Foods
11:13:35 21 had developed a supercrictical CO2
11:13:35 22 extraction to decaffinate coffee.
11:13:35 23 Nicotine and caffeine are both
11:13:38 24 alkaloids. They are sister compounds,
11:13:52 25 they behave in very much the same way.

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11:13:52 So we thought maybe the same technology would be useful for denicotinizing tobacco.

11:13:52 Other things had been used in the past but the problem is, they were not very selective.

11:14:10 These solvent systems would remove other things besides nicotine and generally render the tobacco pretty useless from a taste point of view.

11:14:10 Supercrictical CO2 is more selective. It's not perfectly selective, but it left more of the other solubles in tobacco that we thought contributed to the taste of the product.

11:14:19 So, we really leveraged or piggybacked on the General Foods decaffinated coffee technology.

11:14:28 In fact, we ran some early trials in their pilot plant in Bremen, Germany.

11:14:34 Q. Can you pinpoint that at a period of time?

11:14:43 A. It was around 1998, I would

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11:14:43 say.
11:14:43 Q. And it was prior to the
11:14:45 ramp-up of Next?
11:14:56 A. Yes. These were just pilot
11:14:56 studies to see if it would work.
11:14:56 Q. But Philip Morris was aware of
11:14:56 the supercrictical process prior to the
11:14:56 purchase of General Foods, correct?
11:14:58 A. Presumably.
11:14:58 Q. You had that technology
11:15:02 nomenclature and procedure available to
11:15:06 you.
11:15:06 MR. MUEHLBERGER: Object to
11:15:06 the form.
11:15:06 A. I'm not really sure what
11:15:12 you're --
BY MR. HOPPER:
Q. Is it my use of the word,
"nomenclature" or what?
I'm not where you're coming
from on that one.
11:15:13 MR. MUEHLBERGER: I don't
11:15:13 understand what the question is.
11:15:13 It was so vague and ambiguous to
11:15:13 me.

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11:15:17 BY MR. HOPPER:
11:15:17 Q. You can answer it.
11:15:18 A. I don't understand the
11:15:19 question, either.
11:15:23 If you are asking me did
11:15:23 Philip Morris have knowledge that
11:15:24 General Foods was using supercrictical
11:15:27 CO2 to decaffinate coffee, I would
11:15:36 guess so.
11:15:36 Q. But I'm asking -- what I'm
11:15:36 asking you, to be clear, is you didn't
11:15:36 have to purchase General Foods to
11:15:36 implement that technology?
11:15:37 A. No.
11:15:37 Q. You were cognizant of the
11:15:38 availability of that technology?
11:15:41 A. That's right.
11:15:43 Q. You had it within the corpus
11:15:43 of your R & D department --
11:15:46 A. Yes.
11:15:46 Q. -- that's what I'm asking.
11:15:48 A. And actually, General Foods
11:15:51 didn't own all of the decaffinization
11:15:57 patents. There was -- the Max Planck
11:15:57 Institute in Germany really owned the

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11:15:57 patents and we ended up taking a
11:15:58 license from Max Planck to operate the
11:15:58 process.
11:16:49 Q. After you extracted the
11:16:52 nicotine in this process, before you
11:17:02 reapplied it to the stems, was the
11:17:04 nicotine held in containers, or how did
11:17:08 you contain it? Where did it go?
11:17:12 A. It was dissolved in the CO2.
11:17:15 It was a continuous process.
11:17:18 Q. So it was kept in a gas vapor
11:17:19 phase?
11:17:19 A. Yes.
11:17:21 Q. For how long?
11:17:22 A. I can't recall what the flow
11:17:25 through the loop was, but their
11:17:27 velocities were very high. It was
11:17:27 probably just a matter of seconds
11:17:28 between one vessel and another vessel.
11:17:31 However, this was sort of a
11:17:33 batch continuous operation. There was
11:17:35 a circulation through both beds until
11:17:37 sufficient nicotine was extracted from
11:17:42 the tobacco and deposited upon the
11:17:43 stem.

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11:17:43 Q. And so, as it was extracted
11:17:46 and made its way through the loops in
11:17:51 the vapor phase, did it go on
11:17:51 immediately to application to the stems
11:17:51 at that point or was it held for any
11:17:52 period of time?
11:17:53 A. No. It would be pretty tough
11:17:56 to hold it.
11:17:59 Q. Okay. Because of the
11:18:00 pressure?
11:18:00 A. Well, yes. And you would have
11:18:01 to separate it from the CO2 somehow as
11:18:04 well.
11:18:04 Q. And how does that happen?
11:18:06 A. In this process, it was just a
11:18:08 physical absorption. The stem would
11:18:12 absorb the nicotine preferentially from
11:18:16 the CO2 and hold it in place.
11:18:19 Q. But it was -- when the
11:18:20 vapor was -- I assume it was pumped
11:18:23 through?
11:18:23 A. Right.
11:18:24 Q. Where were the stems contained
11:18:26 at that point?
11:18:27 A. They were in a large pressure

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11:18:30 vessel. So the flow was from the
11:18:34 tobacco vessel, through the vessel,
11:18:35 through some pipes, into the stem
11:18:36 vessel, through the stem, and then it
11:18:38 would return back to the tobacco
11:18:40 vessel, having the nicotine removed or
11:18:43 absorbed by the stem.
11:18:45 So, you had relatively free
11:18:51 nicotine going back for another pass at
11:18:51 removing more from the tobacco.
11:18:52 Q. Would the vapor go into the
11:18:54 vessel on the whole, or would it be
11:18:57 applied in a more of direct fashion
11:19:02 right to the stem?
11:19:02 A. Well, there were distributors
11:19:03 to distribute the flow over the surface
11:19:06 of the vessels. So, you got as good a
11:19:10 flow distribution as you could.
11:19:11 I mean, you don't want to do a
11:19:12 lot of channeling because you would
11:19:15 reduce the efficiency of the process,
11:19:19 make it take longer.
11:19:20 Q. What kind of controls,
11:19:22 sensors, measuring devices, perhaps
11:19:27 manufactured by other companies that

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11:19:31 you had on this equipment, did you use
11:19:33 to measure that reapplication process?
11:19:37 A. We actually had a -- sort of a
11:19:40 home-grown, home-built, nicotine
11:19:43 analyzer in the vapor phase.
11:19:46 So, we could then tell when no
11:19:50 additional nicotine was being removed
11:19:52 from the tobacco because the
11:19:54 concentration of the gas phase was very
11:20:00 low. And we attempted to use that to
11:20:01 control the cycle time.
11:20:03 It turns out, though, you
11:20:05 know, after a short period of time it
11:20:09 was very, very repeatable and just
11:20:09 running for a certain period of time
11:20:12 and then analyzing the alkaloid
11:20:15 concentration at the end of the batch
11:20:17 to assure compliance to the
11:20:22 specification was adequate.
11:20:27 Q. Who manufactures the equipment
11:20:29 that you used for that process?
11:20:33 A. Oh, lots and lots and lots of
11:20:34 people.
11:20:35 The vessels were made by
11:20:39 Tissen in Germany. They are very, very

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11:20:43 high-pressure vessels. The wall
11:20:46 thickness was something like a foot.
11:20:48 And they were made of a very special
11:20:51 stainless steel. The compressors were
11:20:53 made yet by other people. You know,
11:20:57 control valves by yet others.
11:20:58 Lots and lots and lots of
11:20:59 manufacturers of equipment.
11:21:01 Q. Were there sensor devices on
11:21:03 these controls?
11:21:04 A. Well, sure.
11:21:06 I mean, there would be, you
11:21:07 know, temperature and flow, and the
11:21:11 normal type of process control
11:21:16 necessary to regulate a --
11:21:16 Q. Industrial controls?
11:21:16 A. Right.
11:21:17 Q. Timing devices?
11:21:19 A. Timing, sure. Programmable
11:21:23 controllers.
11:21:25 Q. Would these be comparable to
11:21:25 the types of industrial controls and
11:21:31 sensors and devices, perhaps made by a
11:21:31 Honeywell or somebody like that?
11:21:31 A. Sure.

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11:21:33 Q. Do you still have this
11:21:35 equipment?
11:21:36 A. We do.
11:21:37 Q. Do you use it?
11:21:39 A. No.
11:21:39 Q. Where is it?
11:21:40 A. It's sitting on the Park 500
11:21:43 site, mothballed.
11:21:43 Q. Is it in the normal industrial
11:21:48 processes and manufacturing of your
11:21:48 conventional cigarettes?
11:21:51 A. No, it's a separate facility.
11:21:54 Q. Is Park 500 still in
11:21:58 operation?
11:21:59 A. It is, yes.
11:21:[illegible] Q. What do you make there?
11:22:00 A. RL.
11:22:04 Q. But you are telling me that
11:22:08 these vessels and these controls and
11:22:08 these devices are not used in any way
11:22:09 whatsoever in the manufacture of
11:22:11 cigarettes?
11:22:15 A. That's right.
11:22:15 Q. Okay. Well, if I went over
11:22:16 there and looked, I would see they were

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11:22:19 on the shelf, so to speak?
11:22:30 A. Love to show you.
11:22:30 Q. Why don't you use them?
11:22:30 A. We don't have an application for them.
11:22:30 Q. Meaning you don't denicotinized cigarettes any longer?
11:22:30 A. We do not.
11:22:30 Q. Okay. If you tell me, and you did tell me, and you testified to the Jury today that you have a nicotine analyzer on the vapor phase, wouldn't it be accurate to say that you can regulate the amount of the nicotine that would go to those stems?

MR. MUEHLBERGER: Object to the form.

MR. HOPPER: Why? What is it?

MR. MUEHLBERGER: Could you read it back, ma'am.

11:22:30 (Reporter read: Okay. If you tell me, and you did tell me, and you testified to the Jury today that you have a nicotine analyzer on the vapor phase, wouldn't it be

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accurate to say that you can regulate the amount of the nicotine that would go to those stems?)

MR. MUEHLBERGER: Object to the form. I just have no idea of what you're trying to ask.

MR. HOPPER: Okay.

MR. MUEHLBERGER: Vague and ambiguous.

A. If I understand your question correctly, that was the objective.

I mean, it was to remove essentially all of the nicotine from tobacco and make sure that it was deposited on the stem.

And that was the only place it could have gone so the objective was to make sure you get it out of the tobacco and get it on the stem.

BY MR. HOPPER:

Q. And you could control that in that automated manufacturing process?

A. Yeah, you would control it by the length of time you let the loop operate.

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11:23:54 Q. But you testified also that
11:23:56 you had controls and sensors and
11:23:56 devices that control temperature, flow,
11:23:58 timing, concentration?
11:24:01 A. Well, I didn't say
11:24:02 concentration. But concentration in
11:24:05 the sense that we could have controlled
11:24:07 the concentration of nicotine in the
11:24:10 gas, in the CO2.
11:24:12 Q. Correct. You said -- and we
11:24:15 could go back and read it if it would
11:24:17 helpful to you -- that you could
11:24:19 analyze the alkaloid concentration by
11:24:22 those devices?
11:24:23 A. We could analyze the alkaloid
11:24:27 concentration in the supercrictical
11:24:30 CO2.
11:24:30 Q. And ninety-five percent of
11:24:32 that alkaloid was nicotine?
11:24:32 A. Yes.
11:24:33 Q. And in that regard, you could
11:24:35 regulate the concentration of nicotine
11:24:35 going into those stems?
11:24:41 MR. MUEHLBERGER: Object to
the word, "regulate."

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11:24:44 A. If you are asking the question
11:24:45 could we change the length of time that
11:24:49 this cycle operated to put more or less
11:24:54 nicotine onto the stems, sure. And
11:24:59 that is exactly what we were trying to
11:25:17 do.
11:25:17 MR. HOPPER: Let's take a
11:25:19 break.
11:25:19 VIDEOGRAPHER: Going off the
11:25:21 record at 11:25 A.M.
(Off record.)
VIDEOGRAPHER: Returning back
11:34:30 on the record at 11:34 A.M.
11:34:30 BY MR. HOPPER:
11:34:36 Q. Mr. Burnley, in the
manufacturing process that you were
11:34:47 just testifying to the Jury about the
extraction of nicotine, once the vapor
phase was pumped into the vessel
11:34:57 containing the stems and applied to the
11:35:08 stems, what did you do with the stems?
11:35:08 A. We ultimately land-filled the
11:35:08 stems.
11:35:08 Q. You didn't use them?
11:35:10 A. No. We made a couple of

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11:35:12 attempts to see if they could be
11:35:16 incorporated in, I believe, BL at a low
11:35:19 level. But we were never able to get
11:35:23 subjective acceptability from our
11:35:29 expert panel, so we ended up actually
11:35:32 washing the stems to remove the added
11:35:35 nicotine and we treated that in the
11:35:38 waste treatment plant at Park 500,
11:35:43 dried the stems and land-filled them.
11:35:45 Q. So, are you telling me that
11:35:48 the extraction of nicotine all went for
11:35:51 naught?
11:35:51 MR. MUEHLBERGER: Object to
11:35:51 the form.
11:35:51 A. Well, nicotine was a
11:35:51 by-product. I mean, we were trying to
11:35:52 remove nicotine from tobacco and you
11:35:56 have to find a way to dispose of it.
11:36:00 BY MR. HOPPER:
11:36:12 Q. So, you put it on the stems
11:36:12 and then you didn't use the stems. You
11:36:12 didn't use them in the RL process at
11:36:12 all?
11:36:12 A. We didn't.
11:36:14 Q. You've never used them in the

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11:36:19 RL process?

11:36:19 A. Well, there may have been a trial where we tried to incorporate a low-level of them. I can't really remember, it's been a long time now.

11:36:20 But we never did it commercially.

11:36:22 Actually, the stems were an interim solution. If this product had been successful, we wouldn't have had enough stems to use to commercially deposit nicotine on them because we used the stems for other purposes.

11:36:39 We actually developed a water scrubber. It just wasn't ready at the time we built the plant. We designed it, we built the prototype. And the intent was to water scrub the nicotine out of the CO2 and then dispose of the nicotine.

11:37:05 Q. You inject water into the vapor phase and it depletes the nicotine?

11:37:13 A. Sure, it was --

11:37:13 Q. Using the water scrubber?

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11:37:15 A. Absolutely. It was a
11:37:20 countercurrent absorber where water was
11:37:24 sprayed in the top of a tower and the
11:37:28 nicotine ladened gas was
11:37:32 countercurrently contacted with the
11:37:32 water. The nicotine would transfer
11:37:32 from the CO2 to the water. I believe
11:37:32 there is some acid in the water, too,
11:37:35 to help that transfer, to help tie up
11:37:39 the nicotine. And then the water was
11:37:43 collected and we were developing a
11:37:43 process to treat the water and dispose
11:37:46 of it.
11:37:47 Q. I have an interesting question
11:37:50 for you. You seem to know almost
11:37:52 everything about all this. I'm
11:37:54 impressed.
11:37:58 Could you remove the nicotine
11:37:58 back out of the water, if you wanted
11:38:00 to?
11:38:02 A. Never really thought about it,
11:38:04 but presumably you could.
11:38:06 Q. Did you ever, or did Philip
11:38:08 Morris ever attempt to remove the
11:38:10 nicotine back out of the water?

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11:38:12 A. No.
11:38:13 Q. Did you collect or hold or
11:38:14 retain that water?
11:38:16 A. We held it for short periods
11:38:20 of time while it was waiting to be
11:38:22 treated in our waste treatment
11:38:25 facility.
11:38:25 Q. Where did you hold it?
11:38:26 A. In tanks.
11:38:27 Q. But in which facility?
11:38:35 A. It was -- we had a pilot plant
11:38:38 where we were doing this work, also on
11:38:38 the Park 500 site.
11:38:36 Q. Was that Semi-Works?
11:38:40 A. No. This was a separate pilot
11:38:40 plant we built just for the development
11:38:41 of denic, and we were doing the work in
11:38:48 that pilot plant on a small scale.
11:38:58 Q. And so ultimately, you are
11:38:59 telling me, and you are testifying to
11:38:59 the Jury today, that you disposed of
11:39:02 the stems in a landfill?
11:39:13 A. Yes.
11:39:13 Q. Which landfill?
11:39:13 A. It was one operated by

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11:39:13 Shoesmith Brothers in Chesterfield
11:39:15 Country, Virginia.
11:39:20 Q. Do they have records of that
11:39:20 delivery? Do they have the logs?
11:39:21 A. I believe so.
11:39:22 MR. MUEHLBERGER: Object to
11:39:23 the form.
11:39:23 A. I believe they do.
11:39:23 BY MR. HOPPER:
11:39:25 Q. Presently, if I wanted to
11:39:28 subpoena those or ask for those
11:39:30 records --
11:39:30 A. I think --
11:39:34 MR. MUEHLBERGER: Let me
11:39:34 just -- the question is whether you
11:39:36 can could subpoena the records from
11:39:37 a third-party? Is that your
11:39:38 question?
11:39:40 MR. HOPPER: I'm asking him if
11:39:42 he knows if they are still
11:39:44 available, if they still exist.
11:39:44 MR. MUEHLBERGER: If a
11:39:44 third-party still has them?
11:39:49 MR. HOPPER: Yes.
11:39:49 MR. MUEHLBERGER: Object to

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11:39:49 form.
11:39:49 A. I mean, I have no way of
11:39:51 knowing whether they still do or not.
11:39:51 BY MR. HOPPER:
11:39:52 Q. Are you aware of whether they
11:39:56 kept logs of materials coming in and
11:40:00 out? Of the landfill.
11:40:01 A. I can't answer for sure. I'm
11:40:05 sure we have records of shipments.
11:40:05 Q. Were you in charge of that
11:40:08 process?
11:40:08 A. No.
11:40:08 Q. Was someone who worked
11:40:08 underneath you in charge of the
11:40:11 disposal process of the stems?
11:40:13 A. At the time, I don't believe
11:40:15 -- the person would have been Dave
11:40:19 Merrill. At the time I don't think he
11:40:19 reported to me.
11:40:19 Q. Can you spell that for the
11:40:20 Court Reporter, please.
11:40:20 A. Dave Merrill. M-E-R-R-I-L-L.
11:40:25 Q. And he was in charge of the
11:40:27 waste disposal of the stems?
11:40:29 A. Yes. He runs the

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11:40:31 Environmental Compliance Department for the company.

Q. Is he still at Philip Morris?

A. He is.

Q. Were those stems disposed of as a toxic or hazardous waste at the time?

A. I don't remember. That could be why we washed them -- to remove the added nicotine.

Q. How much of the -- I'm sorry?

A. I'm just not sure.

Q. How much of the nicotine was ultimately removed, percentage-wise, would you say?

A. Probably more than we added, simply, because, you know, it's not selective. When you are washing stems, you are going to wash all soluble material off, including some non-nicotine containing compounds.

Probably more than was added.

Q. Did you dispose of the, as you described it, waste water, after the washing?

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11:41:34 A. Yes.
11:41:34 Q. And where did you dispose of
11:41:35 that?
11:41:35 A. We processed it in the Park
11:41:41 500 waste water treatment facility.
11:41:42 Q. Is that a waste water
11:41:43 treatment facility operated by Philip
11:41:46 Morris?
11:41:46 A. Yes.
11:41:47 Q. Is it susceptible and governed
11:41:52 by waste water treatment laws and
11:41:57 regulations that any waste water
11:42:00 treatment facility would govern?
11:42:00 MR. MUEHLBERGER: Object to
11:42:03 the form.
11:42:03 A. Absolutely. We have certain
11:42:05 discharge parameters that we have to
11:42:09 meet.
11:42:09 BY MR. HOPPER:
11:42:09 Q. And you keep logs of
11:42:15 everything that goes in?
11:42:15 A. Yes, we do.
11:42:15 Q. Do you still have the logs of
11:42:16 the waste water from the washing of the
11:42:24 stems for the nicotine extraction

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11:42:27 procedure?
11:42:28 A. I don't know. I don't know
11:42:28 what the retention schedule on that
11:42:29 stuff is.
11:42:29 Q. Do you have to report on a
11:42:31 regular basis to the EPA on that
11:42:36 disposal -- on any disposal to that
11:42:39 facility?
11:42:39 A. I don't know whether it's EPA
11:42:43 or probably the State Water Control
11:42:44 Board.
11:42:45 Q. But to some governmental
11:42:47 authority that might have jurisdiction
11:42:49 over those matters?
11:42:51 A. Absolutely.
11:42:54 Q. Would you have made a report
11:42:55 on those at the time, in compliance --
11:42:58 MR. MUEHLBERGER: Object to
the form.
BY MR. HOPPER:
Q. -- presumably?
A. Are you asking the question
specifically related to washing the
stem?
Q. Yes, sir.

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11:43:04 A. I don't really know.
11:43:04 Q. Mr. Merrill, who would be in
11:43:07 charge of the environmental aspects of
11:43:09 Philip Morris, would know that?
11:43:16 A. Presumably.
11:43:19 Q. Was Philip Morris ever
11:43:20 investigated by any Federal or State or
11:43:26 local governmental authority with
11:43:26 respect to the disposal of those stems?
11:43:29 A. Not to my knowledge.
11:43:31 Q. Were they ever investigated
11:43:33 with respect to the disposal of the
11:43:39 so-called waste water?
11:43:40 A. Not to my knowledge. I mean,
11:43:40 I'm not really that close to that
11:43:46 facility.
11:43:46 Q. Have there been any inquiries
11:43:48 made outside of litigation with respect
11:43:50 to that process or procedure, that you
11:43:57 are aware of?
11:43:57 A. Not that I'm aware of.
11:43:58 Q. Has the media made any
11:43:59 inquires?
11:44:00 A. Not that I'm aware of.
11:44:02 Q. Who would be?

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11:44:03 A. Again, I would refer you to
11:44:10 David Merrill.
11:44:10 Q. All right.
11:44:31 MR. HOPPER: Can we mark this
11:44:33 as Burnley Exhibit Number Three.
11:44:33 (Whereupon, the document was
11:44:3 marked as Exhibit No. 3 for
11:44:42 Identification)
11:44:42 BY MR. HOPPER:
11:44:42 Q. Mr. Burnley, I'm about to show
11:44:46 you, and we will provide copies to
11:44:50 counsel, what I have marked as Burnley
11:44:52 Exhibit Number Three. It's also been
11:44:59 given an exhibit number for purposes of
11:45:00 this litigation as Scott Exhibit Number
11:45:02 3377. And I believe it also retains a
11:45:08 Philip Morris Bates stamp of 0177203
11:45:18 through 01772174.
11:45:25 It's about twelve pages long
11:45:26 and it's considered to be the
11:45:28 motives -- it's titled to be, "The
11:45:29 Motives and Incentives in Cigarette
11:45:34 Smoking." And it appears to be a paper
11:45:34 produced by a William L. Dunn of the
11:45:38 Philip Morris Research Center.

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11:45:40	1	Do you know Mr. Dunn?
11:45:40	2	MR. MUEHLBERGER: Let me
11:45:40	3	object to the exhibit.
11:45:42	4	MR. HOPPER: Sure.
11:45:42	5	MR. MUEHLBERGER: You
11:45:42	6	indicated, I believe, that it
11:45:42	7	contained a Philip Morris Bates
11:45:45	8	number.
11:45:46	9	MR. HOPPER: I believe it
11:45:47	10	does.
11:45:49	11	MR. MUEHLBERGER: And it's my
11:45:49	12	understanding that that was not a
11:45:50	13	Bates Number that was applied by
11:45:50	14	Philip Morris, but other
11:45:51	15	plaintiff's counsel in other
11:45:51	16	litigation.
11:45:54	17	MR. HOPPER: Okay, I stand
11:45:56	18	corrected.
11:45:57	19	Whatever the case may be, I
11:46:03	20	just wanted to be sure, if you had
11:46:03	21	a Philip Morris indication, that
11:46:03	22	that was included in the record.
11:46:03	23	But if that's not the case, I
11:46:05	24	stand corrected
11:46:05	25	BY MR. HOPPER:

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11:46:05 Q. Do you know who William L. Dunn, Jr., is?

11:46:10 A. I have heard his name from time-to-time but I didn't know him.

11:46:11 Q. This was apparently delivered at Williamsburg, Virginia, in October -- just about, almost twenty years ago, October 22nd through the 28th, 1972.

11:46:22 You came to work at Philip Morris in late '73; is that right?

11:46:24 A. That's right.

11:46:28 Q. Was he still working at Philip Morris at the time in R & D?

11:46:33 A. I believe he was. But I just didn't know him.

11:46:37 Q. And I believe, looking back at your C.V., you were a Systems Engineer at that time?

11:46:46 A. Right.

11:46:49 Q. Was that under R & D?

11:46:49 A. No.

11:46:50 Q. Who did you report to at that time?

11:46:50 A. At the time, that was either part of the IS Organization or part of

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11:47:00 the engineering process. It
11:47:00 flip-flopped affiliations for a while,
11:47:03 but it was probably part of the
11:47:03 Engineering Department.
11:47:14 Q. And that was separate from
11:47:14 Research and Development?
11:47:14 A. It was at the time, yes.
11:47:14 Q. Did you ever meet Mr. Dunn?
11:47:14 A. I don't remember. I don't
11:47:16 remember him.
11:47:16 Q. You have heard his name,
11:47:17 though?
11:47:17 A. I have, yes.
11:47:25 Q. I'm going to show you what has
11:47:28 been marked as Burnley Exhibit Number
11:47:31 Three and I'm referring to Page Twelve.
11:47:34 And there are -- I would like
11:47:41 for you to read from this paragraph
11:47:45 here out loud into the record.
11:47:47 If you want to read it to
11:47:50 yourself first, that is fine. And then
11:47:52 down through the conclusion of point
11:47:56 three.
11:48:00 (Tendering document.)
A. Can I look at the context of

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this Exhibit?

Q. Sure, go right ahead. Take as long as you need.

(Perusing document.)

11:48:58 MR. MUEHLBERGER: Let me just
11:48:59 object to the extent you haven't
11:49:03 established this witness has ever
11:49:06 seen this document. He didn't even
11:49:10 recall knowing the author. He was
11:49:17 not even working in the department
11:49:20 at the time. I think it's
11:49:23 inappropriate for you to ask him to
11:49:23 read a portion of a document he's
11:49:23 never seen before into the record.
11:49:23 Mr. Hopper: Well, Number One,
11:49:23 I disagree with you, counsel. I
11:49:23 did establish that. I asked him
11:49:23 the prefatory foundational
11:49:23 questions about it.
11:49:25 He stated that he did not
11:49:25 recall meeting Mr. Dunn. He stated
11:49:28 that he did not work in systems
11:49:31 engineer as a part of R & D, which
11:49:31 is part of your objection.
11:49:32 He stated that he had heard of

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11:49:37 Mr. Dunn and so, I think you are
11:49:39 certainly allowed to enter your
11:49:44 objection on the record but I want
11:49:46 to enter on the record the fact
11:49:55 that I did lay the prefatory
11:49:55 foundation. So, I think he is
11:49:55 capable of answering the questions
11:49:55 and reading into the record what
11:49:55 I've given him from the document.
11:49:55 MR. MUEHLBERGER: As I
11:49:58 understand it, the only foundation
11:49:58 is that he's heard of this man.
11:50:08 MR. Hopper: No, that's not
11:50:08 the only foundation.
11:50:09 He worked at the company at
11:50:09 the time in 1973. He eventually
11:50:09 came to work as a part of the R & D
11:50:12 Department. He collaborated with
11:50:12 the R & D department.
11:50:16 All of this has been placed on
11:50:19 the record considerably in the
11:50:20 early parts of this deposition.
11:50:22 But you certainly are entitled
11:50:22 to object and raise that at a later
11:50:24 stage, if that's necessary.

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11:50:25 MR. MUEHLBERGER: Does it end
11:50:26 at Page Twelve? Because I have two
11:50:26 blank pages in it.
11:50:28 MR. Hopper: Yes, I have two
11:50:28 blank pages, too. It ends at Page
11:50:28 Twelve. I think the copy machine
11:50:28 just spit out a couple extra
11:50:32 copies.
11:50:37 BY MR. HOPPER:
11:50:37 Q. Have you familiarized yourself
11:50:37 enough with the document?
11:50:38 A. Well, let me read it.
11:50:40 Is this a business document?
11:50:42 Q. Yes, it is.
11:50:44 A. Well, it's a very strange
11:50:46 business document.
11:50:46 Q. Well, all I'm asking you to do
11:50:49 right now is not comment on it, but to
11:50:49 read that portion that I asked you to
11:50:51 read.
11:50:52 MR. MUEHLBERGER: Well, he has
11:50:53 asked to review the document since
11:50:55 he has never seen it before.
11:50:58 MR. HOPPER: Right. And I'm
11:51:00 giving him the time to review it.

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11:51:07 THE WITNESS: I'm still reading.

11:51:07 MR. HOPPER: Take your time.

11:51:21 And I guess I would like to ask the witness to refrain from any comments, unless it's made pursuant to a question. And I would object to the witness being nonresponsive for the past comments made. To enter that objection on the record.

11:51:26 THE WITNESS: (Perusing document.)

MR. HOPPER: What I was referring to, counsel, I didn't ask his opinion on the document or ask him to comment on the document as a direct question. So, his comment is nonresponsive. So, I just wanted to be sure to get that objection on the record.

11:52:02 MR. MUEHLBERGER: Let me also object, counsel, to the extent this doesn't bear an official Philip Morris Bates number, and I have no idea whether this is a business

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11:52:21	1	record or not. There has been no
11:52:22	2	foundation laid for it and the mere
11:52:25	3	fact that this witness was employed
11:52:25	4	by another division of the company
11:52:27	5	at the time is certainly not
11:52:29	6	foundation, to the extent you are
11:52:31	7	trying to admit a portion of this
11:52:34	8	document into the record.
11:52:35	9	MR. HOPPER: Excuse me,
11:52:37	10	counsel. If you wouldn't mind to
11:52:40	11	confine your objections to
11:52:43	12	evidentiary objections and not
11:52:45	13	speaking objections in compliance
11:52:47	14	with CMO-2, I would appreciate it.
11:52:49	15	If you objection is that it's
11:52:49	16	not a business record, then that's
11:52:51	17	acceptable.
11:52:53	18	I believe, however, that these
11:52:54	19	documents have all been
11:52:55	20	authenticated by your counsel at
11:52:58	21	Adams & Reese upon providing these
11:53:02	22	pursuant to request for production
11:53:04	23	of documents.
11:53:05	24	MR. MUEHLBERGER: Well, that
11:53:06	25	is not true because you've

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11:53:06 indicated, for instance, that it
11:53:08 bears a Philip Morris Bates number
11:53:08 when, in fact, it doesn't bear a
11:53:09 Philip Morris Bates number.
11:53:09 MR. HOPPER: It may not --
11:53:11 MR. MUEHLBERGER: So, your
11:53:12 statement is completely inaccurate.
11:53:39 A. Well, I have read the
11:53:40 document. I just think the language in
11:53:43 the document and the content --
11:53:47 MR. HOPPER: Excuse me,
11:53:47 Mr. Burnley. I haven't asked you
11:53:51 any questions or asked for any
11:53:52 comments on the document, so I
11:53:52 object to that being nonresponsive.
11:53:56 I'm asking you if you would
11:53:58 read that portion of the document
11:53:58 that I asked you to read, and
11:53:58 that's all I'm asking you to do at
11:54:02 this point, sir.
11:54:02 THE WITNESS: Sure.
11:54:02 MR. MUEHLBERGER: And I will
11:54:02 again raise all of my objections.
11:54:04 This is completely inappropriate.
11:54:04 There is no foundation for this.

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11:54:04 BY MR. HOPPER:

11:54:06 Q. Okay.

11:54:06 A. "The majority of the conferees would go even farther and accept the proposition that nicotine is the active constituent in cigarettes smoke.

11:54:14 "Without nicotine, the argument goes, there would be no smoking.

11:54:17 "Some strong evidence can be marshalled to support this argument.

11:54:25 "Number One, no one has ever become a cigarette smoker by smoking cigarettes without nicotine.

11:54:28 "Number Two, most of the physiological responses to inhaled smoke have been shown to be nicotine related.

11:54:33 "Number Three, despite many low nicotine brand entries into the marketplace, none of them have captured a substantial segment of the market. In fact, critics of the industry would do well to reflect on the indifference of the consumer to the industry's

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efforts to sell low delivery brands.

"Ninety-four percent of the cigarettes sold in the United States deliver more than one milligram of nicotine. Ninety-five percent deliver more than nine-tenths of a milligram.

"The physiological response to nicotine can readily be elicited by cigarettes delivering in the range of one milligram of nicotine.

"I hope our English friends who are developing a synthetic nicotineless cigarette are not going to be too disturbed by all of this."

Q. Thank you.

Mr. Burnley, I'm now about to show you a document which I have marked as Burnley Deposition Exhibit Number Four.

(Whereupon, the document was marked as Exhibit No. 4 for Identification.)

MR. Hopper: And I'm providing copies to your counsel and will give you adequate time to review

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11:56:05 the document and for your counsel
11:56:05 to review it.
11:56:06 And certainly if there are any
11:56:09 evidentiary objections that your
11:56:11 counsel wants to enter on the
11:56:13 record, he or she certainly have
11:56:13 the time and the opportunity to do
11:56:38 that.
11:56:38 (Tendering document.)
11:56:38 BY MR. HOPPER:
11:56:40 Q. In particular, Mr. Burnley, I
11:56:43 would like for you to look at the
11:56:45 second page of this document, which is
11:56:47 a memorandum on Philip Morris
11:57:00 letterhead, and it's dated 1977, when
11:57:00 you worked at Philip Morris, and it's a
11:57:00 memo from Dr. R. B. Seligman to
11:57:02 Mr. T. S. Osdene, and the subject
11:57:04 matter of it is, "Some comments about
11:57:06 the CTR program."
11:57:08 And I would like you to read
11:57:14 to this point where I'm showing you on
11:57:14 the document.
11:57:14 Take the time you need to
11:57:14 review the document.

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11:57:15 MR. MUEHLBERGER: Just so I'm clear, you are going to ask him to again read from a document that you have established no foundation whatsoever?

MR. HOPPER: I believe I have but you can enter your objection that there's no foundation, but that's all you can enter.

Under the rules.

MR. MUEHLBERGER: Well, it's also hearsay.

I don't know what other objections may apply to this document. This is a completely inappropriate line of questioning.

A. (Perusing document.)

So, this document is from Tom Osdene to Bob Seligman, is that what you said?

BY MR. HOPPER:

Q. No, look at the second page. It's from Seligman to Osdene, isn't it?

A. I think this says to and from,

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11:57:58 so it's from Osdene.
11:57:58 Q. Okay, sure.
11:58:02 Do you know Tom Osdene?
11:58:05 A. Yes.
11:58:06 Q. And who was Tom Osdene?
11:58:09 A. Tom had a variety of jobs.
11:58:16 I'm not sure what he did in 1977.
11:58:16 He was a researcher and later
11:58:23 ran part of the research organization
11:58:24 for Philip Morris, USA.
11:58:26 In '77, I'm not sure what his
11:58:32 position was.
11:58:33 Q. And did you know Dr. Seligman?
11:58:34 A. Yes.
11:58:34 Q. And what did Dr. Seligman do
11:58:38 at Philip Morris?
11:58:35 A. Again, in '77, I can't tell
11:58:38 you. He ultimately became
11:58:43 Vice-President of Research.
11:58:39 Q. And you, yourself, came under
11:58:43 the auspices of Research and
11:58:46 Development at a point in time in your
11:58:49 career, too, correct?
11:58:50 A. Yes.
11:58:51 Q. And did you work with Tom

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11:58:53 Osdene and Dr. Seligman at any point
11:59:05 during your career?
11:59:05 A. No.
11:59:05 Q. Did they still work in R & D
11:59:05 when you became a part of R & D?
11:59:05 A. No.
11:59:05 Q. Okay. Had they retired from
11:59:05 the company by then?
11:59:05 A. Yes, they had.
11:59:05 Well, no, Tom Osdene was still
11:59:05 employed by the company but he was in a
11:59:08 different department at the time.
11:59:11 Q. Okay.
11:59:11 A. (Perusing document.)
11:59:26 Would you like for me to read
11:59:28 it?
11:59:38 BY MR. HOPPER:
11:59:38 Q. Please, sir.
11:59:41 MR. MUEHLBERGER: Are you
11:59:41 asking him now to read a November
11:59:45 27th, 1977, document?
11:59:48 MR. Hopper: I have asked him
11:59:49 to read the second page, right
11:59:51 here.
11:59:51 MR. MUEHLBERGER: That

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11:59:52 being -- my question is, there are two memos here.

11:59:54 MR. HOPPER: Yes.

11:59:54 MR. MUEHLBERGER: So, you are asking him to read from the November 29th, memo?

11:59:56 MR. HOPPER: Yes.

11:59:57 MR. MUEHLBERGER: Again, object to the form. Lack of foundation.

11:59:59 MR. HOPPER: Okay.

12:00:02 MR. MUEHLBERGER: Hearsay. It's completely improper.

12:00:02 A. Again, this is a memo from T. S. Osdene to Dr. R. B. Seligman.

12:00:04 The subject is: "Some comments about the CTR program." And the paragraph you asked me to read is as follows:

12:00:08 "I was amazed at the trend that the CTR work is taking. For openers, Dr. Donald H. Ford, a new staff member, makes the following quotes. Opiates and nicotine may be similar in action.

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12:00:30 "We accept the fact that
12:00:31 nicotine is habituating. There is a
12:00:35 relationship between nicotine and the
12:00:35 opiates."
12:00:40 BY MR. HOPPER:
12:00:40 Q. Okay. Thank you.
12:01:15 I'm now about to show you, and
12:01:15 we will also provide copies to your
12:01:15 counsel, what has been marked as
12:01:15 Burnley Exhibit Number 5.
12:01:17 It's also labeled as Scott
12:01:19 Plaintiff's Exhibit 3948.
(Whereupon, the document was
marked Burnley Exhibit No. 5 for
Identification.)
MR. HOPPER: And just to
correct the record, the prior document
that I just gave you, Madam Reporter,
12:01:30 was also marked as Scott Plaintiff's
12:01:30 Exhibit 3483.
12:01:36 I believe I forgot to enter
12:01:40 that into the record.
12:01:40 BY MR. HOPPER:
12:01:42 Q. I'm going to show these copies
12:01:44 to counsel.

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12:01:44 And take the adequate time you
12:01:50 need to review these. But what I'm
12:01:50 interested in having you do is read
12:01:51 this last paragraph here.
12:01:52 The document is correspondence
12:01:56 from Mr. Bill Dunn to Mr. Jack Hind.
12:02:01 And it's on Philip Morris letterhead
12:02:04 from your Research Center, again, and
12:02:12 it's dated March 15, 1978. And notably
12:02:12 there is a copy to Mr. R. B. Seligman.
12:02:12 MR. MUEHLBERGER: Let me just
12:02:22 again object to the extent you're
12:02:23 asking this witness to read into
12:02:24 the record a document for which
12:02:26 there has been no foundation
12:02:34 established.
12:02:34 MR. Hopper: Okay.
MR. MUEHLBERGER: I'm sorry,
counsel, what portion have you
asked him to read?
MR. HOPPER: That last
paragraph on the bottom of the
page, of the first page.
A. (Perusing document.)
12:06:02 I really can't understand the

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12:06:06 context of the letter. But I'm happy
12:06:10 to read the last paragraph.
12:06:10 BY MR. HOPPER:
12:06:10 Q. Thank you.
12:06:14 A. "As far as the nicotine
12:06:14 reinforcement, we are beginning to
12:06:15 suspect that rather than there being an
12:06:19 attempt to maintain some blood
12:06:19 consistency levels, the smoker is
12:06:22 seeking the spike effect of blood
12:06:23 nicotine which occur immediately upon
12:06:26 inhalation.
12:06:28 "These effects may well be so
12:06:30 rewarding that the habitual smoker
12:06:30 overloads himself in seeking them, with
12:06:34 high blood nicotine levels occurring
12:06:38 after the fact and not essential to the
12:06:41 reinforcement process."
12:06:44 Q. Thank you.
12:06:46 Now, Mr. Burnley, you've
12:06:47 observed that this letter was sent from
12:06:50 your Research Center at Philip Morris,
12:06:54 correct?
12:06:54 MR. MUEHLBERGER: Object to
12:06:55 the form. He has no idea.

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12:06:55 BY MR. HOPPER:
12:06:57 Q. You observed the document
12:07:00 itself?
12:07:00 A. It's on Philip Morris
12:07:01 letterhead. It was sent to a retiree
12:07:03 named Jack Hind from Mr. Dunn.
12:07:08 Q. Okay.
12:07:49 Mr. Burnley, earlier in this
12:07:52 deposition, you testified to the Jury,
12:07:54 and you used the word "sensory impact."
12:07:58 What did you mean by the use of that
12:08:01 term?
12:08:01 A. The taste of the product.
12:08:05 Q. Does it take up any other
12:08:07 descriptors or definitions beyond just
12:08:12 taste?
12:08:12 MR. MUEHLBERGER: Object to
12:08:12 the form.
12:08:12 A. Well, when I use the word, I'm
12:08:15 generally referring to the -- how the
12:08:18 product tastes. There is also sort of
12:08:22 a trigeminale aspect of smoking.
12:08:22 BY MR. HOPPER:
12:08:31 Q. Help me with trigeminal,
12:08:31 that's a new one on me.

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12:08:31 A. Well, it's taste and odor.
12:08:31 Q. Okay.
12:08:34 A. Smoke affects the trigeminale
12:08:42 nerve. So, it's whatever the sort of
12:08:42 sensory, taste, smell experience is
12:08:43 that the smoker enjoys when smoking.
12:08:50 Q. Has Philip Morris, in its
12:08:50 Research Department or elsewhere in the
12:08:51 company, conducted any types of sensory
12:08:54 or neurologically related tests to
12:09:01 determine how to calibrate or set taste
12:09:12 and smell and aroma of the cigarettes?
12:09:20 MR. MUEHLBERGER: Object to
12:09:20 the form.
12:09:20 A. Well, you are getting pretty
12:09:20 far outside of my area of expertise.
12:09:20 I'm not aware of any.
12:09:20 BY MR. HOPPER:
12:09:22 Q. Which area of the company
12:09:22 would such research and/or testing be
12:09:22 conducted? Would it be the Flavor
12:09:22 Department?
12:09:26 MR. MUEHLBERGER: Object to
12:09:26 the form.
12:09:26 A. Well, I think I would start

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12:09:27 with the people who are responsible for
12:09:30 product development.
12:09:30 BY MR. HOPPER:
12:09:31 Q. And who do they report to?
12:09:34 MR. MUEHLBERGER: Object to
12:09:34 the form.
12:09:35 A. Today, they report to Urs
12:09:37 Nyffeler, who is the Senior
12:09:42 Vice-President of R & D.
12:09:42 BY MR. HOPPER:
12:09:51 Q. Okay. Well, is that an
12:09:52 objective or a subjective term, as it's
12:09:56 used -- sensory impact?
12:09:59 MR. MUEHLBERGER: Object to
12:09:59 the form.
12:10:02 A. It's a sensory. It's a
12:10:06 descriptor of how the smoker perceives
12:10:10 the product.
12:10:10 BY MR. HOPPER:
12:10:11 Q. Is it subjective or objective?
12:10:21 A. Oh, it's subjective. It's
12:10:21 very subjective.
12:10:22 Q. Does Philip Morris have any
12:10:23 objective standards as to what sensory
12:10:33 impact should be?

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12:10:33 A. The only objective standards may be when trying to match a competitive product in the marketplace, an expert panel may smoke the product and try to describe in more detail than the lay panelists could, what they are tasting. And in certain situations, a leaf blender may know how to put a blend together or may know how to add a flavor system that would tend to match a competitive product.

Q. To achieve the desired subjective taste?

A. Yes.

MR. MUEHLBERGER: Object to the form.

BY MR. HOPPER:

Q. Mr. Burnley, I'm about to show you a document that I have marked as Burnley Exhibit Number 6.

(Whereupon, the document was marked as Exhibit No. 6 for Identification.)

It is a research paper written by T. R. Schori, S-C-H-O-R-I, and it's

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titled, "Smoke Impact from a Psychologist's Vantage Point."

And it was produced pursuant to the Arch V American Tobacco Company litigation, in what I believe later became the Barnes case.

I would like for you to take the time you need to look at the document. It's also marked, excuse me, as Scott Plaintiff's Exhibit Number 3420.

MR. HOPPER: And counsel, take the time you need to look at it.

BY MR. HOPPER:

Q. And I would like for you to read on Page 2 of the document, the highlighted portion.

MR. MUEHLBERGER: I presume the highlighting is yours, counsel?

MR. Hopper: Yes, it is.

MR. MUEHLBERGER: Let me again object. I think it's inappropriate to be --

MR. HOPPER: Excuse me, counsel. Make a specific

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12:12:44 evidentiary objection, if it's form
12:12:47 or if it's no foundation. And no
12:12:50 speaking objections, please.

I just ask you to exercise discretion. I'm trying to be civil about it, too, but --

I'll give you all the time and room you want to make your objection.

MR. MUEHLBERGER: That's what I was trying to do.

MR. HOPPER: Okay.

MR. MUEHLBERGER: I think it's
12:12:16 completely inappropriate to attach
12:12:[illegible] documents --

12:12:[illegible] MR. HOPPER: No, excuse me.
12:13:00 Excuse me.

12:13:00 Time out, counsel.

12:13:00 MR. MUEHLBERGER: Let me
12:13:01 finish what I'm saying.

12:13:01 MR. HOPPER: No, counsel, I'm
12:13:02 not going to allow you to put a
12:13:05 speaking objection onto the record.
12:13:07 We have specific, procedural
12:13:09 rules in Louisiana that are guiding

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12:13:13 this litigation to disallow
12:13:14 speaking objections.
12:13:14 MR. MUEHLBERGER: I'm not
12:13:17 making a speaking objection, I'm
12:13:17 trying to indicate that your
12:13:17 procedure of putting documents on
12:13:20 this record which have no bearing
12:13:20 whatsoever to this witness'
12:13:21 knowledge or foundation --
12:13:24 MR. HOPPER: So, that's your
12:13:28 objection? A lack of foundation?
12:13:27 MR. MUEHLBERGER: A lack of
12:13:28 foundation.
12:13:28 MR. HOPPER: Then state that.
12:13:28 MR. MUEHLBERGER: And it's
12:13:29 inappropriate.
12:13:29 MR. HOPPER: State that and
12:13:30 there's no room under the Louisiana
12:13:31 rules of procedure adopted by the
12:13:37 CMO-2, which you stipulated to,
12:13:37 which has been entered into this
12:13:38 record, that allows you to speak
12:13:40 out that objection and enter into
12:13:40 it and thereby counsel your witness
12:13:43 in any way.

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12:13:44 So, if you want to go and talk
12:13:46 to the Special Master about that,
12:13:50 I'm happy to do it.
12:13:50 I want to be civil about this,
12:13:52 but those are the rules that have
12:13:54 been set down by the Court and by
12:13:59 the Judge in the case.
12:13:59 MR. MUEHLBERGER: Well, I
12:13:59 understand that. This has come up
12:14:00 before and we have talked to the
12:14:00 Special Master about it. He is out
12:14:02 of town unfortunately, is my
12:14:02 understanding, today.
12:14:03 But if we continue doing this,
12:14:05 I'm just putting you on notice, I'm
12:14:05 going to continue to object.
12:14:05 MR. HOPPER: Okay.
12:14:07 MR. MUEHLBERGER: It's
12:14:07 improper. If this is how you spend
12:14:11 your time, at five o'clock, we're
12:14:14 shutting down and we're going home.
12:14:17 You have got thousands of
12:14:18 pages of testimony from this
12:14:19 witness and there is simply no
12:14:19 point in going over documents he

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12:14:19 simply has never seen before and
12:14:20 has no knowledge of.
12:14:20 MR. HOPPER: Is that your
12:14:20 objection?
12:14:25 Are you just lecturing me?
12:14:27 Are you counseling the witness?
12:14:29 And thereby I'm going to -- elicit
12:14:33 an objection from me that counsel
12:14:36 is testifying.
12:14:36 What are you doing? Are you
12:14:39 lecturing me?
12:14:42 MR. MUEHLBERGER: I just made
12:14:42 my objection for the record.
12:14:43 MR. HOPPER: You did not make
12:14:44 -- no, you did not, counsel, make
12:14:44 an objection for the record.
12:14:46 You entered a speaking
12:14:47 objection which is disallowed under
12:14:50 this litigation.
12:14:52 And all I can do is advise you
12:14:54 that the Court has said you cannot
12:14:57 enter speaking objections. I am
12:15:00 not going to allow them and I will
12:15:00 interrupt you in the future if you
12:15:04 continue to do that and we will go

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12:15:04 off the record and I don't mean to
12:15:06 be in any way obstreperous about
12:15:08 that. I want to be sure for the
12:15:15 written record and for the
12:15:15 videotape, that this is being
12:15:15 addressed in a civil manner and
12:15:17 that I'm advising counsel, no
12:15:19 speaking objections.
12:15:21 MR. MUEHLBERGER: Well, let
12:15:21 me say that you're not going to
12:15:24 tell me what I am and am not going
12:15:24 to say on this record. You're not
12:15:24 going to tell me what objections I
12:15:27 am or am not going to make. And
12:15:28 you're not the Judge or Special
12:15:30 Master and you're not going to rule
12:15:31 on them.
12:15:32 MR. HOPPER: No, I am going to
12:15:33 because this is my deposition on
12:15:35 behalf of my clients, not yours.
12:15:38 And I'm going to tell you, and I'm
12:15:40 going to tell you pursuant to what
12:15:45 the Special Master has ruled,
12:15:46 whether you like it or not.
12:15:48 So, we can cut it off there

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12:15:50 and have no more speaking
12:15:52 objections, or we are going to have
12:16:10 to end up calling the Special
12:16:10 Master and call the Judge about it.
12:16:10 MR. MUEHLBERGER: Fine. I'm
12:16:10 going to continue to make my
12:16:10 objections.
12:16:10 MR. HOPPER: Well, then, I
12:16:10 suggest that you make them and
12:16:10 confine them to lack of foundation
12:16:10 or to whatever objection you want
12:16:10 to make.
12:16:10 MR. MUEHLBERGER: That is what
12:16:10 I'm been trying to do.
12:16:10 MR. HOPPER: Well, you haven't
12:16:10 because you have been entering
12:16:14 these running, speaking objections,
12:16:18 using things like, "inappropriate,"
et cetera, which I don't think are
found in the rules of evidence
anywhere, and certainly not in the
rules of evidence in Louisiana.
BY MR. HOPPER:
12:16:19 Q. But having said all that,
12:16:21 Mr. Burnley, would you please read from

http://legacy.library.ucsf.edu/tid/vm07a00/pdf
Support http://www.industrydocuments.ucsf.edu/docs/yfjl0001

12:16:24 Page 2 of the document that I have
12:16:27 provided to you that is marked Burnley
12:16:30 Exhibit Number 6.
12:16:30 MR. HOPPER: Correct, Court
12:16:35 Reporter?
12:16:35 MR. MUEHLBERGER: Same
12:16:36 objections.
12:16:36 A. Okay, I'm reading from a
12:16:38 document by T. R. Schori, entitled,
12:16:41 "Smoke Impact from a Psychologist's
12:16:44 Vantage Point."
12:16:44 And a part of the paragraph
12:16:53 that you asked me to read is as
12:16:53 follows: "Specifically, though, what
12:16:57 component in cigarette smoke is
12:16:59 responsible for activating these
12:16:56 receptors and, thus, for giving rise to
12:16:57 the sensory experiences that we call
12:16:59 smoke impact. At the time that the
12:17:12 sensory model for smoke impact was
12:17:12 first conceptualized (Schori, 1974B), I
12:17:12 was not prepared to hypothesize as to
12:17:12 which specific component of cigarette
12:17:12 smoke caused smoke impact. However, I
12:17:15 am now prepared to hypothesize. It is

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12:17:18 nicotine."
12:17:19 Q. Thank you.
12:17:19 MR. HOPPER: Let's take a
12:17:19 one-minute break while the Court
12:18:28 Reporter changes her tape.
12:18:28 VIDEOGRAPHER: Going off the
12:18:28 record at 12:17 P.M.
12:18:28 (Off record.)
12:18:34 VIDEOGRAPHER: Returning back
12:18:35 on the record at 12:18 P.M.
BY MR. HOPPER:
Q. Mr. Burnley, are you familiar
with the ART cigarette?
A. Yes.
Q. What is the ART cigarette, for
the Jury?
A. It's the same product we were
talking about a few moments ago.
It stands for alkaloid reduced
tobacco or denicotinized cigarette.
Q. Slang, the denic?
A. Yes.
Q. You have used that term?
A. Right.
Q. Okay. Thank you.

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12:19:51 And Next was one of those
12:19:54 products?
12:19:54 A. It was.
12:19:54 Q. Were there other products
12:19:54 developed by Philip Morris that were
12:19:55 denic or ART cigarettes?
12:19:56 A. Yes. There was Merit denic
12:19:57 and probably a few other line
12:20:08 extensions, I just can't recall what
12:20:08 they are called today.
12:20:08 Q. Were those commercially
12:20:08 marketed?
12:20:08 A. They were.
12:20:08 Q. And how long were they
12:20:08 commercially marketed?
12:20:08 A. It varied from test-market to
12:20:08 test-market. But over a period of a
12:20:12 couple of years, we had a number of
12:20:13 test-markets active in different parts
12:20:16 of the country.
12:20:17 Q. And those products similar to
12:20:20 Next were only in a test-market phase;
12:20:25 correct?
12:20:25 A. That's correct.
12:20:44 Q. Have you ever been present at

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12:20:45 meetings at Philip Morris where your
12:20:48 colleagues, or executives, or senior
12:20:52 executives, ever discussed maximizing
12:20:55 the delivery of nicotine in your
12:21:03 products?
12:21:03 A. No.
12:21:03 Q. Is it your conclusion that the
12:21:05 alkaloid or nicotine component of
12:21:11 cigarettes remains constant then,
12:21:15 across cigarettes?
12:21:15 MR. MUEHLBERGER: Object to
12:21:16 the form.
12:21:16 A. Generally speaking, the
12:21:19 concentration of nicotine in smoke
12:21:20 follows the concentration of tar. They
12:21:23 are related.
12:21:23 BY MR. HOPPER:
12:21:24 Q. Does Philip Morris use filler
12:21:30 in its cigarette?
12:21:58 A. Filler is a term to describe
12:22:00 cut tobacco. So, yes, we use it. We
12:22:05 call it cut filler, yes.
12:22:09 Q. Is it distinguished or
12:22:10 different in nature from other tobacco?
12:22:14 A. No. It is the cut total blend

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12:22:18 of tobacco that goes into a cigarette.
12:22:21 Q. Does Philip Morris sell its
12:22:23 products volumetrically?
12:22:26 Is there a volume measured
12:22:28 amount of each component that goes into
12:22:37 a cigarette?
12:22:37 MR. MUEHLBERGER: Object to
12:22:37 the form.
12:22:37 A. Well, cigarettes essentially
12:22:38 are sold volumetrically. There is a
12:22:41 certain circumference and length
12:22:44 specification, and they are filled to
12:22:47 that volumetric specification.
12:22:46 Although each product has its own
12:22:49 cigarette weight specification.
12:22:49 BY MR. HOPPER:
12:22:52 Q. Is that true with respect to
12:22:54 the constituents of cigarettes as well,
12:23:01 and not just the product contents?
12:23:12 A. No. Except to the extent that
12:23:12 flavors are applied as a function of
12:23:12 weight. So, there is a ratio between
12:23:12 the amount of flavors applied and the
12:23:14 amount of weight that is in a
12:23:14 cigarette. So, they are linked.

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12:24:09 MR. HOPPER: I think this may
12:24:11 be a good point to stop for lunch.
12:24:11 * * *
12:24:20 VIDEOGRAPHER: Going off the record
12:24:24 at 12:24 P.M.
* * *
(Following a lunch break, the following procedures were had.)
* * *
VIDEOGRAPHER: Returning back
13:21:52 on the record at 1:20 P.M.
* * *
MR. HOPPER: Madam Court Reporter, would you mind reading back the last question, please.
(Off record while reading computer screen.)
/
/
/
AFTERNOON SESSION
HAROLD BURNLEY, WITNESS
* * *
BY MR. HOPPER:
13:22:28 Q. Mr. Burnley, we are back on

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13:22:31 the record for the afternoon.
13:22:33 And you're aware from the many
13:22:35 depositions you have taken, you are
13:22:37 still under oath?
13:22:39 A. Yes.
13:22:40 Q. We were discussing, and I
13:22:42 asked you a question about the filler
13:22:49 before we took the break.
13:22:49 Do you add other compounds or
13:22:50 anything else to filler when you
13:22:58 manufacture cigarettes?
13:22:66 A. No. As I testified to a
13:23:00 little while ago, filler is a term to
13:23:01 use -- it's used to describe all of the
13:23:04 tobacco components that are cut at
13:23:07 generally thirty cuts per inch to make
13:23:11 filler for cigarettes.
13:23:14 Filler refers to the fact that
13:23:15 it fills the cigarette. It's not any
13:23:19 particular substance.
13:23:20 Q. Okay. There are -- in
13:23:21 addition to the flavors and the
13:23:23 humectants that we discussed earlier
13:23:27 this morning, you answered, I believe,
13:23:29 that you add to cigarettes; are there

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13:23:34 other additives that you add to
13:23:36 cigarettes in the manufacturing?
13:23:39 A. None come to mind, no.
13:23:41 Q. Has Philip Morris ever
13:23:42 added -- strike that.
13:23:47 Are you familiar with
13:23:47 aliphatic organic acids?
13:23:51 A. I have heard the term.
13:23:55 Q. Such as formic acids or
13:23:55 propionic acids?
13:23:55 A. I've heard the terms, yes.
13:24:01 Q. Has Philip Morris ever added
13:24:01 aliphatic organic acids, or formic
13:24:11 acids to any of its products?
13:24:11 A. I don't know. If they did, it
13:24:11 would have to have been as a flavor
13:24:12 system component.
13:24:12 I'm not aware of any.
13:24:19 Q. If formic acid was oversprayed
13:24:23 on the sheets, would that necessarily
13:24:23 increase the nicotine content?
13:24:23 MR. MUEHLBERGER: Object to
13:24:26 the form.
13:24:26 A. I don't see how. There is no
13:24:27 nicotine in formic acid. So, spraying

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13:24:44 formic acid on tobacco couldn't really
13:24:44 increase the nicotine content.
13:24:44 BY MR. HOPPER:
13:24:44 Q. Do you know what substances,
13:24:44 if added to your products, would
13:24:44 increase the nicotine content?
13:24:46 A. Other than adding nicotine?
13:24:52 Q. Yes.
13:24:52 A. I can't think of anything.
13:25:21 Q. You stated in your Statement
13:25:23 of Opinion in your Expert Witness
13:25:28 Report that you submitted as a part of
13:25:31 this proceeding, that one of the things
13:25:33 you would testify to, and I quote, Item
13:25:38 Number 2, and I'm reading.
13:25:38 "The reasons for using ammonia
13:25:42 compounds in Philip Morris'
13:25:42 manufacturing processes."
13:25:44 You just testified a moment
13:25:44 ago that you don't add any other
13:25:47 additives; but, in fact, you're stating
13:25:52 in your opinion that you are going to
13:25:52 testify how and why you have used
13:25:55 ammonias in the manufacture of your
13:25:58 products.

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13:25:59 Is that not inconsistent?
13:26:02 A. I don't think so.
13:26:04 Certain ammonia compounds have
13:26:08 been used as flavors for RL.
13:26:10 Also, we do add, still do
13:26:12 today, diammonium phosphate and
13:26:17 ammonium hydroxide as a processing aid
13:26:18 in the manufacture of DL.
13:26:20 In fact, what happens is,
13:26:24 diammonium phosphate in the presence of
13:26:29 ammonia reacts with calcium pectate,
13:26:34 which is naturally occurring in
13:26:34 tobacco, and releases it. Makes it
13:26:37 water soluble so it can then be used as
13:26:37 a, sort of a natural glue, or a natural
13:26:37 binder to hold the tobacco pieces
13:26:40 together in the formation of a sheet.
13:26:46 Q. Has Philip Morris ever used
13:26:48 ammonium bicarbonate in the process of
13:26:52 tobacco, manufacturing its products?
13:26:54 A. Yes. I believe ammonium
13:26:57 bicarbonate was added to cut tobacco in
13:27:03 the denic process, to aid in the rate
13:27:07 at which nicotine is extracted from
13:27:11 filler.

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13:27:11 Q. Now, you testified earlier
13:27:14 today that you studied chemistry
13:27:14 extensively. And certainly as a
13:27:14 chemical engineer, you employ your
13:27:17 knowledge of chemistry in your job.
13:27:19 And you stated in the opinion of your
13:27:24 expert report, that you're relying on
13:27:25 your knowledge in a given field.
13:27:27 Are you aware that in 1997, in
13:27:35 other Castano-related cases,
13:27:39 specifically the Small case in New York
13:27:42 and the Barnes case in Philadelphia, in
13:27:44 deposition testimony that you testified
13:27:47 that you had knowledge of the fact that
13:27:53 when ammonium bicarbonate or other
13:27:55 ammonias are added, that they create
13:27:59 toxic and hazardous fumes such as
13:28:03 nitrogen oxide, NO2, and NH3?
13:28:05 A. Say that again, please.
13:28:05 Q. Are you aware of the fact that
13:28:09 in 1997, in two other Castano -- I call
13:28:20 them companion cases to this case --
13:28:20 the Small case in New York and the
13:28:20 Barnes case in Pennsylvania, that in
13:28:22 deposition testimony, you testified

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13:28:23 that you were aware that toxic fumes
13:28:34 such as nitrogen oxide, and NO2, and
13:28:34 NH3, are emitted when ammonium
13:28:37 bicarbonate is added to your products?
13:28:39 A. I don't recall that.
13:28:39 If you could read it back to
13:28:39 me, maybe it would refresh my memory.
13:28:42 Q. I don't have the transcript.
13:28:42 A. I don't recall making a
13:28:48 statement like that.
13:28:48 Q. Are you aware that that
13:28:48 occurs?
13:28:48 A. No.
13:28:48 Q. Do you believe that it occurs?
13:28:50 A. No.
13:28:51 Q. Do you believe that there are
13:28:55 nitrogen oxides and NO2s and NH3s that
13:29:01 are released in your products when
13:29:02 pyrolyzed?
13:29:05 A. Yes. There is some potassium
13:29:09 nitrate present in cigarette filler.
13:29:09 Q. Are you aware that the EPA has
13:29:13 labeled NOX, and NO2, and NH3 as
13:29:13 extremely hazardous substances?
13:29:15 A. I know that certain public

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13:29:17 health authorities have been concerned
13:29:19 by nitrogen oxides.
13:29:23 I have not heard, at least in
13:29:25 the concentrations that we are using,
13:29:27 that NH3 or ammonia is considered a
13:29:32 hazardous substance.
13:29:34 Q. Are you aware they have been
13:29:38 labeled by EPA as such?
13:29:38 A. Well, I suspect if you're
13:29:42 carrying a tank car of it or something
13:29:43 like that, yes. You wouldn't want to
13:29:43 breathe a concentrated dose of ammonia.
13:29:47 You would suffocate.
13:29:47 Q. If you broke it down to parts
13:29:53 per million, with your knowledge of
13:29:53 chemistry, do you have an idea of how
13:29:53 many parts per million that a person
13:29:54 would need to be exposed to before it
13:29:54 would be toxic?
13:29:59 A. No, I'm not sure.
13:29:59 Q. You think it would be a small
13:30:00 amount?
13:30:01 A. Probably not.
13:30:01 MR. MUEHLBERGER: Object to
13:30:01 the form.

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13:30:04 MR. HOPPER: Pardon me?
13:30:04 MR. MUEHLBERGER: Object to the form.
13:30:05 MR. HOPPER: What is it? He is being produced here as an expert
13:30:06 today. I think I can question him
13:30:14 on that.
13:30:14 MR. MUEHLBERGER: Your
13:30:14 question used the word, "small
13:30:14 amount." I don't have any idea
13:30:18 what you meant by that.
13:30:22 MR. HOPPER: Well, I qualified
13:30:22 it as parts per million though.
13:30:22 MR. MUEHLBERGER: I didn't
13:30:22 hear that part of the question.
13:30:22 MR. HOPPER: Oh.
13:30:22 MR. MUEHLBERGER: Sorry.
13:30:22 MR. HOPPER: That's okay.
13:30:22 A. I mean, I don't know. OSHA
13:30:26 and the American Association of
13:30:29 Industrial Hygienist has published
13:30:30 toxicological limits for a lot of
13:30:33 substances, including ammonia, and I
13:30:37 just don't know what they are off the
13:30:39 top of my head.

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13:30:39 BY MR. HOPPER:

13:30:41 Q. Did you study toxicology when you were preparing to be a chemical engineer?

13:30:43 A. No.

13:30:43 Q. Did you study chemistry to the extent that you know that certain chemicals are toxic to humans at certain levels, parts per million?

13:30:55 A. Sure.

13:30:55 Q. Did you ever read or study Doulls Toxicology?

13:30:57 A. No.

13:30:58 Q. Are there any labels on your products that you produce at Philip Morris that tells a consumer that NOX, NO2 and NH3 are substances that they are exposed to?

13:31:12 A. No.

13:31:17 Q. Are you aware that when ammonium bicarbonate is used as an additive to tobacco stems that it acts as an acid, that it creates nicotine?

13:31:30 A. To my knowledge, we don't add ammonium bicarbonate to stems.

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13:31:37 Q. Have you ever?
13:31:37 A. Not to my knowledge. I can't
13:31:39 recall.
13:31:42 Q. Have you ever added other
13:31:45 ammonia compounds?
13:31:46 A. Sure.
13:31:46 Q. As those the ones that you
13:31:48 cited to a moment ago?
13:31:50 A. Yes.
13:31:50 Q. And the purpose of that would
13:31:52 be to what?
13:31:55 A. Well, there are two purposes.
13:31:55 On our BL, or blended-leaf process,
13:31:57 they are used to convert calcium
13:31:59 pectate to ammonia pectate so that the
13:31:59 pecta molecule can be used as a glue,
13:32:03 or binder, to hold the tobacco pieces
13:32:10 together.
13:32:10 In the case of RL, some
13:32:11 diammonium phospate is used to produce
13:32:11 a subjectively similar tasting product
13:32:15 to BL.
13:32:22 Q. Okay. Now, you say that would
13:32:22 be your intended purpose. And as you
13:32:22 referenced it in your expert report,

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13:32:22 stating that you would testify as to
13:32:26 the reasons for using ammonia compounds
13:32:27 in the manufacturing processes, that
13:32:30 would be why you would use them?
13:32:32 A. Yes.
13:32:32 Q. Are you aware that there are
13:32:40 any residual effects from the use of
13:32:40 those ammoniates, or ammonia compounds?
13:32:40 A. I have heard a lot of
13:32:42 allegations over the past couple of
13:32:45 years.
13:32:45 To the best of my knowledge,
13:32:45 and I have discussed it at length with
13:32:48 my colleagues, we don't believe -- the
13:32:52 company doesn't believe -- that there
13:32:57 is any human pharmacology effect due to
13:33:01 the ammonia compounds that we use at
13:33:02 the levels we use.
13:33:03 Q. Are you aware in the
13:33:04 manufacturing processes in using the
13:33:07 ammonium compounds, that in addition to
13:33:16 creating the effect of a binder, as you
13:33:17 say, that there are other residual
13:33:20 effects that occur as a result of those
13:33:23 ammonia compounds being introduced?

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13:33:23 MR. MUEHLBERGER: Object to
13:33:26 the form.
13:33:26 A. You would have to tell me what
13:33:28 they are.
13:33:28 BY MR. HOPPER:
13:33:29 Q. Well, I'm asking you first if
13:33:31 you know, or it's your understanding in
13:33:34 your experience there are residual
13:33:36 effects?
13:33:37 I say residual. I use that
13:33:41 word because you tell me your purpose
13:33:42 is to create binders for the tobacco.
13:33:42 A. That's right.
13:33:48 Q. And that's your intent. And
13:33:51 that is your testimony pursuant to your
13:33:54 expert report; correct?
13:33:54 A. Yes.
13:33:55 Q. Are there other residual
13:33:57 effects that you know of?
13:33:57 A. Well, I think what happens,
13:33:59 and what has happened historically, is
13:33:59 the company began using BL, which
13:34:02 contained a certain amount of added
13:34:04 ammonia. It was a result of pectin
13:34:07 release reaction. And over time, the

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13:34:11 use of those compounds became a part of
13:34:16 the subjective character of the
13:34:16 product. It became part of the taste
13:34:20 of the product.
13:34:21 In the mid-70's, the company
13:34:23 decided to build additional
13:34:26 reconstituted tobacco capacity, and it
13:34:33 elected to build the papermaking, or RL
13:34:35 technology.
13:34:35 This product smoked
13:34:37 differently from BL. So, as we began
13:34:53 introducing it into our products, the
13:34:54 flavor chemists tried to match, to the
13:34:54 best of their ability, the taste of BL.
13:34:54 Never were completely successful.
13:34:54 So, they began added DAP to RL
13:34:54 to try to make these two substances
13:34:59 similar so that the consumer would find
13:35:00 the product modification acceptable.
13:35:05 Q. Tell the Jury what DAP is so
13:35:05 that they know and understand this.
13:35:05 A. DAP is an acronym that stands
13:35:42 for diammonium phosphate.
13:35:42 Q. Do you have any knowledge, or
13:35:42 information, or experience that adding

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13:35:46 these various ammonium compounds also
13:35:49 increases the level of nicotine in your
13:35:53 products?
13:35:54 A. It really can't increase the
13:35:59 level of nicotine.
13:35:59 I mean, the only way you could
13:36:09 increase the level of nicotine is to
13:36:00 add additional nicotine and these
13:36:02 ammonium compounds do not contain
13:36:06 nicotine.
13:36:07 So, it really can't increase
13:36:10 the concentration of nicotine.
13:36:15 Q. Do they reinforce the PH of
13:36:18 nicotine?
13:36:19 MR. MUEHLBERGER: Object to
13:36:21 the form.
13:36:21 A. I don't understand the
13:36:22 question.
13:36:22 BY MR. HOPPER:
13:36:25 Q. Do they reinforce the
13:36:27 so-called kick that a smoker gets from
13:36:30 nicotine?
13:36:30 MR. MUEHLBERGER: Object to
13:36:31 the form.
13:36:31 A. You know, I have heard the

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13:36:35 word, "kick" many times but I don't
13:36:35 really know what it means. I mean, I
13:36:37 have asked our subjective experts what
13:36:41 kick means and you get a variety of
13:36:45 answers from a variety of people.
13:36:46 I don't know what the term
13:36:47 means. And I don't know how to equate
13:36:50 it to any particular chemistry.
13:36:51 Q. Kind of like addiction? You
13:36:54 don't know what that means, either?
13:36:57 A. Well, I have my own sort of
13:36:59 sense for what addiction means.
13:36:59 Q. Okay.
13:37:14 When I questioned you earlier
13:37:14 in the deposition about sensory impact,
13:37:20 you gave your understanding of that as
13:37:23 being something related to taste,
13:37:26 smell, and I forget the third piece of
13:37:31 your triangle.
13:37:33 A. Taste and smell.
13:37:39 Q. Do you believe that when
13:37:39 ammonia compounds are introduced into
13:37:44 your products, that you get a sensory
13:37:49 impact from that introduction?
13:37:50 A. I think so. I mean, I think

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13:37:52 ammonia plays a role in the taste of
13:37:57 the product. I do.
13:38:28 Q. Are you familiar with, in the
13:38:30 same vein as sensory impact, the term
13:38:36 used in your industry with respect to
13:38:38 reinforcing agents?
13:38:42 MR. MUEHLBERGER: Object to
13:38:42 the form.
13:38:42 A. I have heard the term.
13:38:42 BY MR. HOPPER:
13:38:44 Q. Have your scientists ever
13:38:48 studied or undertaken any research with
13:38:51 respect to reinforcing agents?
13:39:04 A. I don't know.
13:39:19 Q. I'm about to show you a
13:39:19 document I have now marked as Burnley
13:39:21 Exhibit Number 7. And it is a
13:39:22 memorandum from Victor DeNoble to
13:39:27 Dr. J. L. Charles.
13:39:28 Do you know either of those
13:39:30 gentlemen?
13:39:30 (Whereupon, the document was
13:39:30 marked Burnley Exhibit No. 7 for
13:39:31 Identification.)
13:39:31 A. I know --

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13:39:31 Q. I shouldn't say gentlemen;
13:39:31 Dr. Charles could be a woman.
13:39:34 A. I do know Dr. Charles.
13:39:42 I have heard of Victor DeNoble
13:39:42 but I don't really know him.
13:39:42 Q. Do you know what the initials
13:39:45 "J. L." Stand for with respect to
13:39:49 Dr. Charles?
13:39:50 A. Jim something.
13:39:51 Q. Are you familiar at all with
13:39:53 any behavorial pharmacology research
13:39:58 that was undertaken at your company in
13:40:00 the Research Department in the '80s?
13:40:01 A. I don't recall any.
13:40:03 Q. You worked there then, right?
13:40:04 A. I did.
13:40:06 Q. In fact, were you not a Chief
13:40:08 Process Engineer at the time?
13:40:09 A. Probably so.
13:40:10 Q. That is what you are telling
13:40:12 me anyway.
13:40:13 I'm going to have you look at
13:40:15 this document, and in particular, I
13:40:47 would like for you to look on Page 2 in
13:40:47 the next to the last paragraph that is

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13:40:47 marked 1-A.

We are going to provide your counsel with copies of these and I would like for you to read that.

It's from your research department at Philip Morris.

MR. MUEHLBERGER: I'm sorry. Just so I'm clear what the question is, you want him to read the whole document?

MR. HOPPER: No, no. He can look at it if he wants. If you would flip over one more page, 1-A, that paragraph.

MR. MUEHLBERGER: You're asking him to read this into the record?

MR. HOPPER: Yes, please.

MR. MUEHLBERGER: Well, I'm going to object to that.

MR. HOPPER: Okay.

MR. MUEHLBERGER: The document speaks for itself. There is no point -- you can ask him questions about the document, but I'm not

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13:40:53 going to allow him to just read
13:40:55 portions into the record.
13:40:55 MR. HOPPER: Well, he can read
13:40:55 it.
13:40:55 MR. MUEHLBERGER: Well, no,
13:40:59 I'm going to instruct him not to
13:40:59 read it.
13:40:59 MR. HOPPER: Are you going to
13:41:00 instruct him not to answer it?
13:41:00 MR. MUEHLBERGER: That's
13:41:03 right.
13:41:03 MR. HOPPER: On what basis?
13:41:03 The basis for instructing a
13:41:06 witness not to answer is only if it
13:41:08 violates attorney-client privilege
13:41:10 or it's in violation of some
13:41:10 protective order. I don't see how
13:41:12 you can do that.
13:41:13 MR. MUEHLBERGER: The basis
13:41:13 is, the document speaks for itself.
13:41:13 You've established no
13:41:13 foundation for it for use with this
13:41:16 witness.
13:41:16 If you want to ask questions
13:41:18 about the document with the

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witness, fine. But he is not just going to just read it into the record with absolutely no basis or foundation.

You might as well read it into the record if that's your point.

MR. HOPPER: I'll be happy to read it into the record.

MR. MUEHLBERGER: Fine, then do so.

MR. HOPPER: I have laid the foundation for the last stream of about a dozen or more questions, and questions that were asked earlier this morning with respect to the extraction of nicotine, with respect to sensory impact.

There is so much ample foundation laid in this deposition to ask these questions, I think quite honestly you don't want it to be entered, which I can understand that, but it has nothing to do with the lack of foundation.

But you have made your

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13:41:59 objection and I respect that.
13:42:01 MR. MUEHLBERGER: The problem
13:42:01 is, you haven't asked him any
13:42:04 questions about the document.
MR. HOPPER: I'm going to.
MR. MUEHLBERGER: You're
asking him to read a document he's
never seen.
MR. HOPPER: I have asked him
questions -- I just held the
document up and I asked him
13:42:08 questions about the authors. I
13:42:10 asked him questions about the
research department.
MR. MUEHLBERGER: Fine.
MR. HOPPER: She can go back
and read them.
MR. MUEHLBERGER: That's fine.
13:42:13 MR. HOPPER: And I want him
13:42:14 to read it and then I'm going to
13:42:17 ask him further questions.
MR. MUEHLBERGER: Oh, you're
not asking him to read it into the
record?
MR. HOPPER: I haven't asked

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him to read it into the record.

MR. MUEHLBERGER: Okay, I'm sorry. I misunderstood. I thought you had asked him to read this paragraph into the record.

MR. HOPPER: No, no, no. I want him to read it, but I haven't asked him to read into the record yet.

MR. MUEHLBERGER: Fine.

MR. HOPPER: And the document and the questions that I have asked, and am going to continue to ask him, relate to the foundational questions that I have just been asking him.

MR. MUEHLBERGER: So, there is no question pending right now? Just so I'm clear.

MR. HOPPER: There is no question pending.

A. (Perusing document.)

Okay. I have sort of scanned it.

BY MR. HOPPER:

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13:46:28 Q. All right. In the context of
13:46:31 the questions that I have asked you
13:46:32 earlier and just a few moments ago
13:46:44 regarding sensory impact, I had asked
13:46:44 you if you are aware of any reinforcing
13:46:44 agents being added to your products.
13:46:47 And are you, in fact, aware of
13:46:50 any reinforcing agents being added to
13:46:54 your products?
13:46:56 A. I'm not aware of anything
13:47:00 being added to the product for the
13:47:05 purpose of a reinforcing agent.
13:47:05 Q. Are you aware of the fact,
13:47:08 having looked at this document from
13:47:11 your research department, that your
13:47:11 company undertook, in the context of
13:47:11 behavioral pharmacology, research on
13:47:11 acetaldehyde as a nicotine reinforcing
13:47:15 agent?
13:47:15 A. That is what it says.
13:47:17 Q. Are you aware of the use of
13:47:19 acetaldehyde or other aldehydes as
13:47:33 reinforcing agents in your products?
13:47:33 A. I'm not aware of any.
13:47:33 Q. Have you ever included any of

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13:47:33 those in the production processes that
13:47:33 you have been involved with?
13:47:36 A. I have not.
13:47:36 Q. Has anyone at Philip Morris?
13:47:36 A. I don't know.
13:47:36 Q. Do you know if any
13:47:36 acetaldehydes or other aldehydes have
13:47:37 been used as reinforcing agents to
13:47:40 boost nicotine?
13:47:41 A. Not to my knowledge.
13:47:43 Q. You have no knowledge
13:47:45 whatsoever of that ever happening?
13:47:47 A. There are aldehydes in
13:48:00 tobacco, but I'm not aware of anyone
13:48:04 adding acetaldehyde or any other
13:48:07 substance for the purpose of
13:48:09 reinforcing nicotine.
13:48:12 Q. Mr. Burnley, I'm about to show
13:49:04 you a document marked as Burnley
13:49:08 Exhibit 8 to this deposition. And it
13:49:13 is a memorandum dated September 8,
13:49:14 1976, on Philip Morris letterhead.
13:49:18 It's interoffice correspondence from
13:49:20 K. S. Burns to Dr. R. B. Seligman. I
13:49:26 believe you testified earlier today you

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13:49:29 knew Dr. Seligman.

13:49:29 (Whereupon, the document was marked Burnley Exhibit No. 8 for Identification.)

13:49:30 A. Yes.

13:49:30 BY MR. HOPPER:

13:49:30 Q. And are you familiar with K.S. Burns?

13:49:41 A. Yes.

13:49:41 Q. Who is K. S. Burns?

13:49:41 A. Ken Burns.

13:49:41 Q. What was his job at Philip Morris?

13:49:41 A. What time? What is the date?

13:49:41 Q. At the time of this memo, in September of 1976.

13:49:42 A. I don't know what his job was.

13:49:44 Q. Was he in the Research Department?

13:49:50 A. He was in the Research Department. He was an engineer.

13:49:50 Q. Was he a Chemical Engineer like you?

13:49:51 A. No, I don't think so.

13:49:51 Q. Did he ever work for you?

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13:49:53 A. He did.
13:49:54 Q. And what was his job working
13:49:55 for you?
13:49:57 A. He was manager of Development
13:49:59 Engineering.
13:50:01 Q. Why would he be corresponding
13:50:03 with Dr. Seligman, do you suspect?
13:50:07 MR. MUEHLBERGER: Object to
13:50:07 the form.
13:50:14 A. I don't know.
13:50:14 BY MR. HOPPER:
13:50:14 Q. Was he under your management?
13:50:14 A. Not in 1977, no.
13:50:15 Q. '76?
13:50:15 A. Or '76.
13:50:15 Q. All right.
13:51:01 I would like for you to look
13:51:11 at the second page of this exhibit and
13:51:11 read what is a memo, again interoffice
13:51:11 correspondence from Mr. P. M. Gauvin to
13:51:17 a Mr. L. P. Meyer in 1977, just a
13:51:22 little over a year later from this
13:51:26 first memo. I would like for you to
13:51:29 take a look at it. It's very short. I
13:51:31 would like for you to read that

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13:51:36 paragraph, if you would, please, to
13:51:36 yourself.
13:52:00 MR. MUEHLBERGER: Are you just
13:52:01 asking him to read it to himself?
13:52:04 MR. HOPPER: To himself, yes.
13:52:04 A. (Perusing document.)
13:52:47 MR. MUEHLBERGER: Counsel, for
13:52:48 the record, I assume these are just
13:52:50 two documents you've attached and
13:52:50 they were not produced to you in
13:52:52 this manner of being attached?
13:52:54 MR. HOPPER: That's my
13:52:54 understanding.
13:52:59 MR. MUEHLBERGER: Okay.
13:52:59 MR. HOPPER: No, I believe
13:53:01 they come under the Scott exhibit
13:53:06 number. Both of those were
13:53:11 produced under that exhibit number.
13:53:17 MR. MUEHLBERGER: Well, the
13:53:17 problem is that they weren't
13:53:19 produced by Philip Morris together
13:53:21 because the Bates number aren't
13:53:31 consecutive.
13:53:31 MR. HOPPER: I understand. I
13:53:31 saw that. One is 18 and one is 24.

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13:53:31 MR. MUEHLBERGER: Right. So, it looks like someone has put them together, but it was not Philip Morris.

13:53:38 A. Okay.

BY MR. HOPPER:

13:53:56 Q. One memo from Gauvin to Meyer says that, "as a follow-up to my memo of 9/21/77 entitled, 'Increased Nicotine in Smoke, low-delivery cigarettes.' I'm enclosing a survey conducted by Ellen Moore of in-house work on methods for increasing nicotine in smoke. The addition of nicotine citrate to the filler appears to have been the primary method for increasing nicotine in smoke."

Was your company studying ways to increase nicotine?

MR. MUEHLBERGER: Object to the form.

13:54:27 A. Well, that is what the memo says.

13:54:30 You know, if you go back in the history of the debate on smoking

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13:54:35 and health, you will find a school of
13:54:38 thought which says that by adding
13:54:42 nicotine, by increasing the nicotine
13:54:48 delivery of smoke, people will smoke
13:54:49 less and, therefore, be exposed to less
13:54:52 tar.

13:54:53 It's a concept that public
13:54:55 health authorities in various parts of
13:54:57 the word have advocated.

13:55:01 There is no doubt in my mind
13:55:04 that the company looked at this kind of
13:55:08 research and evaluated it. But I've
13:55:10 got to tell you, to the very best of my
13:55:10 knowledge, the company has never added
13:55:12 nicotine, at least in any measurable
13:55:13 quantities, to the product.

13:55:18 And the only exceptions to
13:55:20 that might be some of the nicotine
13:55:23 sulphate that was used as an alcohol
13:55:24 denaturant for flavor systems. And
13:55:27 probably some minor amounts of nicotine
13:55:33 that were a result of tobacco extracted
13:55:35 from--- flavor systems extracted from
13:55:40 tobacco.

13:55:41 But to my knowledge no, no

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13:55:41 appreciable quantities of nicotine have
13:55:41 ever been added to any Philip Morris
13:55:47 product.
13:55:47 That's not to say somebody in
13:55:49 a research and development organization
13:55:51 didn't look at it.
13:55:53 Q. You have no knowledge of
13:55:56 nicotine citrate being added to filler?
13:55:59 A. Commercially, no, not at all.
13:56:15 Q. Why would your company be
13:56:18 studying and researching so
13:56:19 prodigiously to increase the level of
13:56:21 nicotine in your products?
13:56:22 MR. MUEHLBERGER: Object to
the form.
13:56:26 A. Well, I don't know that they
13:56:26 were doing it in a prodigious manner.
13:56:28 I mean, you have got a memo,
13:56:40 and there may be others, where people
13:56:40 looked at adding nicotine to the
13:56:40 product for exactly the reasons I
13:56:40 mentioned awhile ago.
13:56:40 There was a time when public
13:56:40 health authorities were really
13:56:42 recommending that cigarette companies

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13:56:43 add nicotine to the product so that
13:56:47 consumers would smoke less and be
13:56:50 subject to less of the hazards
13:56:54 associated with whole smoke.
13:56:56 But again, to my knowledge it
13:56:57 was never, ever commercialized.
13:57:42 BY MR. HOPPER:
13:58:09 Q. Mr. Burnley, in your past and
13:58:09 current job, do you know if your
13:58:10 company has ever been concerned with
13:58:14 the issue of nicotine safety?
13:58:24 A. Yes.
13:58:24 Q. In what respect?
13:58:24 A. Primarily, with regard to the
13:58:24 denic plant that we discussed at length
13:58:24 earlier.
13:58:24 We had high pressure CO2
13:58:29 containing nicotine circulating. And
13:58:34 that CO2 contained quite a bit of
13:58:39 nicotine. And if there were a leak,
13:58:38 for example, it could be a very serious
13:58:43 safety situation.
13:58:43 Q. Is that because nicotine is
13:58:44 considered to be one of the few liquid
13:58:46 natural alkaloids that is highly toxic

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13:58:50 and highly poisonous?
13:58:52 A. Pure nicotine in sufficient
13:58:56 quantities is highly toxic.
13:58:58 Q. Is it toxic in your products?
13:59:00 A. I don't believe so.
13:59:01 Q. Why?
13:59:02 A. The concentrations are low.
13:59:03 Q. Is it up on -- at a certain
13:59:09 level of consumption and infusion into
13:59:10 the bloodstream by a smoker?
13:59:18 A. I don't understand your
13:59:18 question.
13:59:18 Q. That one can up-take a certain
13:59:18 level of nicotine that it becomes toxic
13:59:22 to them?
13:59:27 MR. MUEHLBERGER: Object to
13:59:27 the form.
13:59:27 A. I have never heard that. But
13:59:27 I'm not an expert either.
13:59:27 BY MR. HOPPER:
13:59:28 Q. Is it poisonous if it's not
13:59:31 toxic?
13:59:32 MR. MUEHLBERGER: Object to
13:59:32 the form.
13:59:33 A. To me they are synonymous

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13:59:38 terms.
13:59:38 BY MR. HOPPER:
13:59:38 Q. Is it addictive?
13:59:40 A. I think by today's definition
13:59:43 of addiction, nicotine is addictive.
13:59:48 Q. Has your company denied in the
13:59:50 past that nicotine was addictive?
13:59:53 A. Well, you know, you have to
13:59:54 look at what point in time you're
13:59:54 talking about.
13:59:55 This definition of addiction,
13:59:57 as you well know, has been evolving
14:00:01 over the last couple of years. Even my
14:00:06 own personal view, and it is just a
14:00:08 personal view because I'm certainly not
14:00:11 an expert, to me, nicotine is not a
14:00:14 highly addictive substance. Certainly
14:00:19 not in the concentrations that most
14:00:22 smokers find in cigarettes.
14:00:22 I mean, I have been a smoker.
14:00:24 I have stopped smoking several times.
14:00:27 People can quit smoking.
14:00:30 Surely, nicotine plays a role.
14:00:32 There is certainly some nicotine
14:00:39 pharmacology going on with smokers.

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14:00:40 But I don't consider nicotine to be one
14:00:44 of those severely addictive drugs as
14:00:46 people sometimes claim.
14:00:48 Q. But it wouldn't surprise you
14:00:49 that any number of the high-level
14:00:53 employees in your company at any given
14:00:55 time, or high-level researchers or
14:00:58 scientists in your company at any given
14:01:02 time have expressed an opinion that
14:01:04 nicotine is highly addictive and that
14:01:10 is why we sell cigarettes, would it?
14:01:11 A. It wouldn't surprise me, no.
14:01:13 Q. And I could probably dump so
14:01:15 many documents in front of you today,
14:01:16 although your counsel would probably
14:01:18 have a coronary if I did, so I won't --
14:01:19 he's a good fellow -- but that says
14:01:22 something completely contrary to what
14:01:24 you are saying, that the company has
14:01:25 know for many, many years that nicotine
14:01:28 is addictive; isn't that true?
14:01:31 A. Look, I tried to give you my
14:01:33 personal view.
14:01:33 Q. I understand but now you're
14:01:33 testifying here as an expert witness

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today and I'm not asking you for your personal view, I'm asking you and examining you as an expert witness.

MR. MUEHLBERGER: Object to the form. Asked and answered.

I'm not sure the question is pending right now, is it?

MR. HOPPER: It is pending but, see, he has answered it with a preface of saying it's his personal view. But he's here testifying as an expert.

MR. MUEHLBERGER: Not as a psychiatrist. Are you asking about addiction? He doesn't have to testify --

MR. HOPPER: He understands the component constituents and everything that goes into the process of making cigarettes. He has been there for thirty years --

THE WITNESS: Twenty-seven.

MR. HOPPER: Twenty-seven, excuse me. Little liberal.

So he knows and understands a

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14:02:13 cigarette. He has stated that in
14:02:19 his expert report.
14:02:19 So, that's the basis of me
14:02:20 examining him.
14:02:20 MR. MUEHLBERGER: Asked and
14:02:20 answered, then.
14:02:22 MR. HOPPER: Well, he has
14:02:23 answered it personally. He even
14:02:23 said it's his personal view.
14:02:25 Now I'm asking you to answer
14:02:31 it as an expert witness, as a
14:02:36 person with great knowledge,
14:02:36 experience, as you have stated
14:02:38 here.
14:02:38 MR. MUEHLBERGER: Object to
14:02:40 the form. Beyond the scope.
14:02:40 Asked and answered.
14:02:44 MR. HOPPER: Well, he hasn't
14:02:46 answered it. He has answered it
14:02:48 personally.
14:02:50 MR. MUEHLBERGER: Well, you
14:02:51 can ask a question but I object to
14:02:55 the extent you're directing this.
14:02:55 MR. HOPPER: Would you like me
14:03:00 to go back and read what he said?

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14:03:01 MR. MUEHLBERGER: No.

MR. HOPPER: Because he said, I'll give you my personal view on it.

Now we've gotten his personal view so I'm asking a different question.

BY MR. HOPPER:

14:03:03 Q. In your capacity of an expert
14:03:06 witness on behalf of the defendant in
14:03:08 this lawsuit.
14:03:10 A. I can't answer questions about
14:03:12 addiction for the company as an expert
14:03:15 witness because I'm not qualified to do
14:03:17 that. It's not my field.
14:03:20 Surely there are documents
14:03:21 where certain people in the company
14:03:23 have said nicotine is addictive. I
14:03:28 have seen them.
14:03:26 I can't speak on behalf of the
14:03:28 company or what the company's position
14:03:30 was as a function of time because I
14:03:33 just don't know what it was or don't
14:03:35 know what definition of addiction would
14:03:38 be referenced at any particular time.

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14:03:41 I'm just not an expert. It's just
14:03:45 beyond my expertise.
14:03:47 Q. Does the fact that nicotine
14:03:48 has remained in your products and that
14:03:51 you have sold your products with
14:03:53 nicotine, helped you sell cigarette?
14:03:55 A. I think so. I think nicotine
14:03:59 and nicotine pharmacology are part of
14:04:01 the smoking experience.
14:04:03 Q. And that it's habituating and
14:04:06 that's helped you sell cigarettes?
14:04:11 MR. MUEHLBERGER: Object to
14:04:11 the form.
14:04:11 A. I don't know what the reason
14:04:11 is.
14:04:11 BY MR. HOPPER:
14:04:27 Q. Mr. Burnley, a moment ago you
14:04:48 testified -- and we can read this back
14:05:47 if you need it to be read, or if your
14:05:50 counsel needs it to be read back
14:06:02 accurately -- that public health
14:06:02 officials were claiming that people
14:06:02 would smoke less if there was perhaps
14:06:02 an increase in nicotine, and that's
14:06:07 perhaps what was motivating your

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14:06:10 Research Department to study and do
14:06:10 research on this topic.
14:06:14 What time period are you
14:06:16 referring to?
14:06:18 MR. MUEHLBERGER: Object to
14:06:18 the form.
MR. HOPPER: The fact that I
asked him to pinpoint this in time
is -- I don't get it.
MR. MUEHLBERGER: To the
extent you mischaracterized his
testimony.
MR. HOPPER: Well, I said we
would read it back if you prefer,
14:06:22 counsel. I'm trying not to take
14:06:22 the time.
14:06:24 He said it a minute ago and
14:06:28 I'm trying to see if you understand
14:06:28 or know what time period you are
14:06:31 referring to.
14:06:32 A. I don't recall.
14:06:33 I have heard on a couple of
14:06:35 instances that there were people in the
14:06:38 United States who had that theory at
14:06:41 one time.

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14:06:42 There was, I think, a belief
14:06:44 by some U. K. Scientists that it was
14:06:48 maybe a way to tackle the smoking and
14:06:53 health problem as well. But I can't
14:06:55 put it in any sort of chronological
14:06:59 perspective for you.
14:06:59 BY MR. HOPPER:
14:07:00 Q. You're not taking it out of
14:07:03 the Surgeon General's Report, though,
14:07:06 for this country?
14:07:07 A. No.
14:07:08 Q. Okay. Any other leading
14:07:29 health authorities you can point to?
14:07:29 A. I can't recall the context but
14:07:32 I've heard it a couple of times. It
14:07:35 seemed to me -- well, that would be
14:07:38 speculating, I don't want to do that.
14:07:42 Q. Okay.
14:07:43 Is this a full and complete
14:07:50 record that you have submitted as a
14:07:53 part of your expert witness report of
14:07:56 your professional and work experiences?
14:08:03 A. Yes.
14:08:03 Q. There is nothing missing here;
14:08:11 correct?

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14:08:11 A. (Witness shakes head.)
14:08:11 Q. So, other than the four years you were at Allied Chemical and the two years you were at Dupont, you have spent the majority of your professional experience, as you refer to it, at Philip Morris, correct?
14:08:25 A. Yes.
14:08:26 Q. When you were at Allied Chemical, what did you do there?
14:08:31 A. I was a Process Engineer. I ran a small mechanical development group, I guess was my actual last assignment there.
14:08:42 Q. Is that different than your job as a Systems Engineer when you came into Philip Morris in '73?
14:08:47 A. Yeah, very much so. I was always interested in applying digital computers to industrial processes, and at that time Philip Morris had an interest.
14:08:54 Through a mutual friend, I got connected with the company and changed jobs.
14:08:58
14:08:59
14:09:02

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14:09:02 Q. And what did you do at Dupont
14:09:07 from '67 to '69?
14:09:07 A. I was a Process Engineer there
14:09:09 as well.
14:09:09 Q. So, you did largely the same
14:09:13 thing at Dupont that you did at Allied?
14:09:13 A. Yes.
14:09:16 Q. Were you aware of the health
14:09:23 effects associated with smoking in '73,
14:09:26 when you came to Philip Morris?
14:09:28 A. Well, I guess I have always
14:09:32 been told that smoking is not
14:09:32 particularly good for you.
14:09:34 Certainly, I had a general
14:09:37 understanding and general belief when I
14:09:40 went to work for the company.
14:09:42 Q. Did it disturb you at all that
14:09:44 the products that you would be in
14:09:46 charge of manufacturing, particularly
14:09:48 as you moved up the ranks to become a
14:09:53 Vice-President of those operations, had
14:09:58 grave health effects on the people who
14:09:58 used those products?
14:09:59 A. Well, I guess I would have to
14:10:01 say that when I joined the company, my

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14:10:04 knowledge of the health effects of
14:10:09 cigarette smoking was pretty vague.
14:10:09 I mean, I had sort of a
14:10:12 common-sense view that smoking
14:10:14 cigarettes probably were not good for
14:10:16 you. My parents taught me that when I
14:10:19 was a kid.
14:10:20 When I joined Philip Morris,
14:10:22 as cigarettes are today, they were a
14:10:26 legal product. It was a very
14:10:31 interesting place to work. Had some
14:10:34 extremely good people. A lot of
14:10:38 technical challenges. And as I spent
14:10:43 more and more time at the company and
14:10:47 really learned more about the health
14:10:51 risks, I spent a lot of time trying to
14:10:55 encourage the company to work in areas
14:10:58 that would help mitigate the risk, to
14:10:59 the extent that is possible.
14:11:02 So, I feel pretty good about
14:11:07 my experience with Philip Morris.
14:11:07 Q. But by and large, your advice
has kind of fallen on deaf ears, hasn't
it?
MR. MUEHLBERGER: Object to

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14:11:11 the form.
14:11:11 A. I don't think so. I don't
14:11:12 think so at all.
14:11:12 BY MR. HOPPER:
14:11:12 Q. In your submission for your
14:11:13 expert witness retention, you have
14:11:18 included a paper, I believe you
14:11:22 authored, on plug flow. "Design and
14:11:25 Application of an upward Plug Flow
14:11:25 Ventilation System."
14:11:22 Aside from the fact that this
14:11:30 paper relates to installing this system
14:11:34 at a opera hall or an art venue of some
14:11:41 sort, can you explain to me what this
14:11:46 has to with your retention as an expert
14:11:50 witness at Philip Morris for
14:11:53 manufacturing and process development
14:11:53 of cigarette products.
14:11:53 MR. MUEHLBERGER: Object to
14:11:56 the form.
14:11:57 A. Probably nothing.
14:11:59 MR. MUEHLBERGER: Object to
14:12:00 the form. I explained that to you.
14:12:00 MR. HOPPER: Whoa. He was
14:12:05 answering my question. He said,

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14:12:05 "probably nothing."

I want to be sure that's on the record.

BY MR. HOPPER:

Q. Isn't that what you said?

A. Yes.

14:12:06 Q. Okay, why did you submit it?

14:12:10 A. It was submitted in my behalf by Hunton and Williams, I assume.

14:12:17 Q. So, you conferred with your attorneys about it?

14:12:21 A. Yes.

14:12:21 Q. Okay.

14:12:22 A. I think there was a request to attach whatever publications I had.

14:12:22 MR. MUEHLBERGER: Yeah, didn't you ask us to do that, counsel? To provide you with publications?

14:12:24 A. That was the only one I had.

14:12:26 MR. MUEHLBERGER: I note for the record that you have requested that.

14:12:34 MR. HOPPER: I probably did, I am not denying it. I'm asking why he submitted that particular paper

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14:12:34 if it had nothing to do with the
14:12:40 retention of his expert witness --
14:12:40 MR. MUEHLBERGER: Because you
14:12:41 asked for publications.
14:12:41 BY MR. HOPPER:
14:12:44 Q. Is that the only thing that
14:12:46 you have published?
14:12:47 A. Yes.
14:12:50 Q. Has that gone through any peer
14:12:51 review, scientific peer review?
14:12:55 A. No. It was actually published
14:13:01 in the proceedings of an in-house
14:13:06 quality conference.
14:13:06 Q. Have you conferred with any
14:13:10 Jury consultants throughout the course
14:13:14 of your preparation for the testimony
14:13:15 today?
14:13:16 A. No.
14:13:16 Q. What about with respect to any
14:13:19 other lawsuits that you have testified
14:13:20 in?
14:13:23 A. No.
14:13:23 Q. Have you conferred with any
14:13:23 kind of outside consultants in
14:13:34 preparation for delivering your

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14:13:34 testimony?
14:13:34 A. I have been in a number of
14:13:34 prep sessions with lawyers who have
14:13:34 been defending my deposition --
14:13:35 defending our company -- but that's
14:13:35 all.
14:13:36 Q. Have you conferred with, or
14:13:37 met with, or been counseled, or coached
14:13:43 by any type of speech coaches or
14:13:44 anything like that?
14:13:45 A. No.
14:13:45 Q. Have you been a part of any
14:13:49 preparations with your lawyers on
14:13:52 any mock cross-examinations?
14:13:56 MR. MUEHLBERGER: Object to
14:13:56 the form. You are talking about
14:13:57 ever? Ever since he's ever
14:13:57 testified in any case?
14:13:58 MR. HOPPER: I'm talking about
14:13:58 with respect to this lawsuit.
14:13:58 MR. MUEHLBERGER: Okay.
14:13:59 A. No.
14:13:59 BY MR. HOPPER:
14:14:01 Q. You didn't practice any
14:14:02 cross-examinations or practice taking

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14:14:04 any questions from your lawyers?
14:14:04 A. No.
14:14:47 Q. Mr. Burnley, you submitted
14:14:50 documents, various document that you're
14:14:54 claiming to rely upon for the purposes
14:14:56 of your testimony today and in this
14:14:59 proceeding and at trial.
14:15:01 I'm marking that as Burnley
14:15:04 Exhibit Number 9.
14:15:04 (Whereupon, the document was
14:15:04 marked Burnley Exhibit Number 9 for
14:15:08 Identification.)
14:15:08 MR. HOPPER: Included is a
14:15:07 memo from an E. Chambers to V. Willis,
14:15:10 Re: Cigarettes for Special Study, dated
14:15:15 6/17/98.
14:15:18 Why do you rely on this
14:15:18 document.
14:15:19 A. In the past, I have been
14:15:21 questioned a lot about the effect of
14:15:24 added ammonia on smoke PH, and the
14:15:28 effects of added ammonia on nicotine
14:15:34 delivery.
14:15:34 And that is simply a memo
14:15:41 describing a test where our products

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14:15:41 were made with and without added
14:15:43 ammonia. And the nicotine delivery and
14:15:47 the smoke pHs are tabulated there.
14:15:56 Q. In this document that you're
14:16:03 referring to, this memo from Elizabeth
14:16:05 Chambers to Vivian Willis, there is a
14:16:09 diagram in here. And included in this
14:16:13 diagram appears to me to be a
14:16:15 calculation of pounds of alkaloids and
14:16:18 pounds of nicotine.
14:16:20 A. That is from a different
14:16:21 document.
14:16:22 It's not related to the Vivian
14:16:25 Willis-Chambers memo.
14:16:32 Q. This is not a part of that?
14:16:34 A. No.
14:16:35 MR. MUEHLBERGER: Look on the
14:16:36 index, Randy. You will see that's
the next document listed.
MR. HOPPER: The Park 500
Process 30-Day Material Balance?
MR. MUEHLBERGER: Yes.
THE WITNESS: Yes.
BY MR. HOPPER:
14:16:42 Q. Excuse me. This is a

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14:16:46 one-pager, then?
14:16:46 A. Yes.
14:16:46 Q. So, this refers to the next --
14:16:48 excuse me. It's a two-pager.
14:16:50 So, the next item is the Park
14:16:50 500 Process 30-Day Material Balance
14:16:55 dated 7-15-1996. And that refers to
14:17:00 this diagram, among other things,
14:17:03 correct?
14:17:03 A. Right.
14:17:04 Q. How is it that you derived at
14:17:08 this calculation of pounds of alkaloids
14:17:12 and pounds of nicotine?
14:17:14 A. Well, very early in some of
14:17:21 the lawsuits, especially in conjunction
14:17:25 with ABC, there were allegations that
14:17:29 the company added nicotine to its RL
14:17:32 product. And I testified a number of
14:17:34 times that really quite the contrary
14:17:37 happened -- that there were losses of
14:17:37 alkaloids and thereby losses of
14:17:38 nicotine during the manufacturing
14:17:40 process.
And this was a special study
that I commissioned to substantiate

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14:17:45 what I had been testifying about.
14:17:51 Q. Isn't it true, though, that
14:17:54 this is labeled here as influent and
14:18:00 not effluent? So, it's not technically
14:18:04 lost, is it?
14:18:11 A. That is influent to the waste
14:18:11 water treatment plant, or effluent from
14:18:11 Park 500.
14:18:11 Q. Okay. Isn't it true that you
14:18:11 testified earlier to me, though, that
14:18:11 you collected and contained those
14:18:18 alkaloids in nicotine in containers?
14:18:21 A. That was related to the denic
14:18:23 project.
14:18:27 Q. But it's not related to Park
14:18:27 500?
14:18:28 A. No.
14:18:29 Q. You don't contain or hold that
14:18:31 product -- the alkaloids or the
14:18:32 nicotine? Is it pumped right out?
14:18:55 A. It's pumped right into the
14:18:55 waste treatment plant, yes.
14:18:55 Q. Okay.
14:18:55 Wny is it that you relied on
14:18:56 as a part of your expert retention, a

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14:18:59 letter from Helmut Gaisch to R.B.
14:19:05 Seligman in September of '83?
14:19:07 A. There has been a lot of
14:19:07 testimony in the litigation about why
14:19:08 the company made a decision to stop
14:19:12 working on a project to dinitrate Park
14:19:18 500 liquor streams using naturally
14:19:21 occurring microorganisms.
14:19:24 I have testified a number of
14:19:26 times that the process just didn't
14:19:36 work.
14:19:36 In that particular memo, it
14:19:36 shows a collaborating point of view by
14:19:36 the then head of R & D in Neuchatel,
14:19:41 Switzerland, the PM Europe.
14:19:50 Q. And why is it that you have
14:19:52 relied on a report by Mr. Uydess and
14:20:00 Dunn on the, "Mixed Culture
14:20:00 Denitrification: Routine Storage and
14:20:00 Start-up Procedures for Refrigerated
14:20:02 Denitrified Seed"?
14:20:04 I assume it is the same thing
14:20:09 you were just now saying, except this
14:20:09 refers to seed?
14:20:10 A. Yes. It just describes some

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14:20:12 of the difficulties that we were having
14:20:15 making this process work commercially.
14:20:15 Q. Who is R. L. Dunn?
14:20:17 A. He was a researcher at Philip
14:20:21 Morris R & D.
14:20:21 Q. Is it the same Dunn that we
14:20:24 referred to earlier in the testimony?
14:20:26 A. I would guess so, yes.
14:20:29 Q. So, you are using work from
14:20:35 Mr. Dunn to rely on for purposes of
14:20:39 your expertise in this case?
14:20:40 A. Well, Dunn and others.
14:20:43 MR. MUEHLBERGER: Let me just
14:20:43 object for the record.
14:20:43 I believe the prior memo was
14:20:44 by William Dunn. This is R. L.
14:20:44 Dunn.
14:20:44 BY MR. HOPPER:
14:20:46 Q. Well, I'm asking. I don't
14:20:48 know.
14:20:48 A. Yeah, I don't really know
14:20:49 either.
14:20:50 Q. Okay. In that particular
14:20:53 document -- excuse me.
14:21:05 In the next document that is

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14:21:16 -- excuse me. In that document -- it's
14:21:18 a little hard to keep track of this
14:21:21 because of the dates -- there is a list
14:21:25 of persons on the distribution list.
14:21:28 Who was M. Hauserman?
14:21:31 A. Max Hauserman. He was
14:21:33 probably the Vice-President of R & D
14:21:35 during that time period.
14:21:37 Q. In '83?
14:21:46 A. Yes.
14:21:46 Q. Did you know him?
14:21:46 A. Yes.
14:21:46 Q. Did you work with him?
14:21:46 A. Yes.
14:21:46 Q. Did you report to him?
14:21:46 A. No.
14:21:48 Q. Did you report to Ken Houghton
14:21:49 at the time; or Houghton?
14:21:51 A. Not at that time, but I knew
14:21:53 Ken Houghton as well.
14:22:09 Q. Who is William Farone?
14:22:09 A. Bill Farone at the time was,
14:22:09 I believe, Director of Applied
14:22:09 Research.
14:22:09 Q. And L. Turano?

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14:22:09	1	A. Lou Turano was Director of
14:22:09	2	Process Development during that period
14:22:12	3	of time.
14:22:12	4	Q. So, he worked for you?
14:22:12	5	A. He did not, no. I was in the
14:22:14	6	Engineering Department during that
14:22:16	7	time.
14:22:16	8	Q. You hadn't moved over to
14:22:18	9	process development then?
14:22:20	10	A. No.
14:22:21	11	Q. And did you know Mr. Farone?
14:22:23	12	A. Yes.
14:22:24	13	Q. Did you work with him?
14:22:25	14	A. Yes.
14:22:27	15	Q. And Mr. Meyer -- Mr. Turano;
14:22:27	16	did you know him and work with him?
14:22:27	17	A. Yes.
14:22:28	18	Q. And then an L. Meyer?
14:22:31	19	A. Yes, Leo Meyer.
14:22:31	20	Q. And what did he do?
14:22:32	21	A. He was Director of Product
14:22:35	22	Development.
14:22:35	23	Q. And did you work with him?
14:22:37	24	A. I did.
14:22:37	25	Q. And Tom Osdene, I believe you

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14:22:40 stated earlier in testimony that you
14:22:42 knew him and worked with him in
14:22:42 research?
14:22:45 A. Yes.
14:22:45 Q. That's correct?
14:22:46 A. I knew him but I had never
14:22:46 really worked closely with Tom.
14:22:47 Q. And a W. Mutter?
14:22:49 A. Walt Mutter, yes, I worked
14:22:53 with him.
14:22:55 Q. And what did he do?
14:22:58 A. He was a research engineer.
14:22:59 Q. In the Research Department in
14:23:03 R & D?
14:23:03 A. Actually, I think he worked in
14:23:03 the Process Development Department.
14:23:03 Q. And what about Cliff Lilly?
14:23:04 A. Cliff Lilly, I'm not sure what
14:23:10 his job was at the time. He was
14:23:10 probably manager of Applied Physics.
14:23:10 Q. In the R & D Department?
14:23:10 A. Yes.
14:23:10 Q. And did he report to Cathy
14:23:10 Ellis?
14:23:10 A. Not during that period of time

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14:23:18 but he did at one point, yes.
14:23:20 Q. And E. Craze?
14:23:23 A. Ed Craze, yes. He was also a
14:23:24 development engineer.
14:23:26 Q. And did you know him and work
14:23:30 with him?
14:23:31 A. I did.
14:23:31 Q. And W. Hempfling?
14:23:33 A. Yes. Walter Hempfling.
14:23:36 Q. And what did he do?
14:23:37 A. He was a microbiologist.
14:23:40 Q. And he was in R & D?
14:23:41 A. Yes.
14:23:42 Q. And S. Hutcheson?
14:23:42 A. Susan Hutcheson was also in
14:23:44 R & D. She had more of an
14:23:46 administrative job at the time.
14:23:49 Q. And Y. Malik?
14:23:53 A. I know the name but I never
14:23:55 really worked closely with him.
14:23:55 Q. That's a man?
14:23:58 A. Yes.
14:23:58 Q. And what did he do?
14:24:00 A. He was a researcher, probably
14:24:01 a microbiologist.

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14:24:04	1	Q. In R & D?
14:24:11	2	A. Yes.
14:24:11	3	Q. And J. Banyasi.
14:24:11	4	A. Banyasi. Physical Chemist.
14:24:11	5	Also worked in R & D.
14:24:15	6	Q. And you knew him?
14:24:16	7	A. Yes.
14:24:17	8	Q. And you worked with him?
14:24:18	9	A. Yes.
14:24:20	10	Q. And E. Bravo?
14:24:23	11	A. Ernie Bravo. I knew who he
14:24:28	12	was, I never really worked directly
14:24:31	13	with him.
14:24:25	14	Q. P. Kusel? K-U-S-E-L.
14:24:29	15	A. Doesn't ring a bell.
14:24:31	16	Q. S. Temhet, T-E-M-H-E-T, it
14:24:34	17	looks like.
14:24:35	18	A. S. Tennet? I don't --
14:24:39	19	Q. Oh, that's T-E-N-N-E-T. I
14:24:39	20	see.
14:24:42	21	A. I know a Joe Tennet, but
14:24:45	22	not --
14:24:45	23	Q. Okay, a D. Chadick?
14:24:52	24	A. Debbie Chadick,
14:24:52	25	Microbiologist.

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14:24:52 Q. In R & D?
14:24:52 A. R & D, yes.
14:24:52 Q. And you knew her and worked with her?
14:24:53 A. Yes.
14:24:54 Q. And P. Oglesby?
14:25:00 A. I don't know "P".
14:25:00 Q. And I assume you are familiar with Central File?
14:25:01 A. Yes.
14:25:02 Q. All right.
14:25:02 You also have included what appears to be an annual report from April of '83, a report by I. L. Uydess. Why did you rely on that? It's titled, "Project 1904."
14:25:19 A. These are all reports on the progress of the naturally occurring denitration project.
14:25:31 And as you follow the reports, you will find that there are times when there was a great deal of excitement. People thought there was a high probability of success. You read the next report and you'll find that more

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problems were discovered.

And this thing was pretty cyclical. It went on for a long period of time.

They are just sort of arbitrarily picked out of a large volume of reporting on NOV. They were just very typical of what was going on during the time.

Q. And you did say denitrification and not denic, right?

A. Denitrification.

Q. Okay.

A. Well, I meant to say denitrification if I said denicotinization.

Q. And that would include a memo from K. H. Cho regarding Project Cancellation on the NOD Plant - Park 500?

A. Yes.

Q. And a memo from J. C. Gomes to H. G. Burnley, that would be you, Jr., excuse me, Re: Status Report on the NOD Project?

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14:26:35 A. Yes.
14:26:36 Q. Same vein?
14:26:37 A. Same vein.
14:26:38 Q. And then there are two patents
14:26:38 listed for a Method of Preparing a
14:26:38 Reconstituted Tobacco Sheet Employing a
14:26:45 Pectin Adhesive and a Tobacco Sheet
14:26:45 Material patent.
14:26:46 Why do you rely on those?
14:26:47 A. They are the patents that
14:26:51 describe the DL Process.
14:26:53 Q. Are those Philip Morris owned
14:26:56 patents?
14:26:57 A. Yes.
14:26:58 Q. Did you confer with or discuss
14:27:01 any of these reliance documents with
14:27:06 your counsel in preparing this report?
14:27:06 A. No.
14:27:07 Q. Did you choose them yourself?
14:27:09 A. Yes.
14:27:09 MR. HOPPER: Let's take a
14:27:09 five-minute break.
14:36:12 (FOLLOWING A BRIEF RECESS, THE
14:36:12 FOLLOWING PROCEEDINGS WERE HAD:)
14:36:12 VIDEOGRAPHER: We are

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14:36:32 returning back on the record at
14:36:34 2:36 P.M.
14:36:34 BY MR. HOPPER:
14:36:37 Q. Mr. Burnley, we are going to
14:36:40 proceed, and as you know you are still
14:36:43 under oath.
14:36:44 A. Yes.
14:36:44 Q. You say you've been employed
14:36:47 with Philip Morris for 27 years?
14:36:49 A. That's correct.
14:36:[illegible] Q. Give or take a few months.
14:36:[illegible] And I believe, if my count is
14:36:49 correct, approximately nine different
14:36:51 positions, something to that effect?
14:36:53 A. Yes.
14:36:54 Q. Can you tell the Jury, because
14:36:58 you are testifying here as an expert
14:37:01 witness today, you are being
14:37:02 compensated. How much are you being
14:37:04 compensated for this testimony?
14:37:07 A. I'm not at all.
14:37:08 Q. You are compensated by the
14:37:11 company, aren't you?
14:37:12 A. My normal salary, yes.
14:37:12 Q. What is that?

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14:37:15 A. Is that your question?
14:37:15 Q. Yes, sir.
14:37:16 A. I can't tell you precisely. It's probably in the neighborhood of two hundred and twenty-five thousand dollars a year.
14:37:25 Q. That's salary and bonuses?
14:37:25 A. That's straight salary.
14:37:27 Q. And then do you receive bonuses on top of that?
14:37:28 A. Depending upon how the company does. How the company believes I do.
I can, yes.
14:37:35 Q. You are in an executive compensation type program, I would assume, as an officer?
14:37:40 A. Yes.
14:37:41 Q. And you are eligible for performance bonuses as well?
14:37:44 A. Yes.
14:37:45 Q. Do you own stock in the company?
14:37:48 A. I do not.
14:37:49 Q. Has the company ever given you stock options?
14:37:52

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14:37:52	1	A. Yes.
14:37:52	2	Q. You own stock options?
14:37:58	3	A. I do.
14:37:58	4	Q. Have you exercised any of
14:37:58	5	those stock options?
14:37:58	6	A. Yes.
14:37:58	7	Q. How much stock option do you
14:38:00	8	hold at this time? How many shares?
14:38:03	9	A. I don't know. I really don't
14:38:05	10	know. I haven't counted it up in a
14:38:08	11	while.
14:38:08	12	Q. If we wanted you to produce
14:38:10	13	evidence of that, you could go to
14:38:21	14	records for us and show us?
14:38:21	15	A. Sure.
14:38:21	16	Q. We'll let your counsel know if
14:38:21	17	we want to receive that.
14:38:21	18	And do you hold preferred
14:38:21	19	stock at all?
14:38:21	20	A. No.
14:38:21	21	Q. Do you hold stock in any kind
14:38:23	22	of employee pension fund that is
14:38:26	23	contributed on your behalf?
14:38:29	24	A. Yes.
14:38:29	25	Q. 401K or some program like

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14:38:29 that?
14:38:29 A. Yes.
14:38:29 Q. Do you know how much stock is
14:38:34 held in there on your behalf?
14:38:34 MR. MUEHLBERGER: You're
14:38:35 saying Philip Morris stock?
14:38:35 BY MR. HOPPER:
14:38:35 Q. Yes, Philip Morris, USA.
14:38:37 A. Oh, Philip Morris stock. No,
14:38:37 there is no Philip Morris stock in
14:38:38 there.
14:38:38 Q. You don't ever receive any
14:38:41 contributions into that pension program
14:38:44 in the way of Philip Morris stock?
14:38:45 A. No.
14:38:58 Q. Do you have any type of
14:39:02 confidentiality agreement that you have
14:39:04 entered into with your employer?
14:39:07 A. Yes.
14:39:07 Q. With respect to trade secrets
14:39:11 and confidential information?
14:39:11 A. Yes.
14:39:11 Q. And you signed that when you
14:39:13 came to work?
14:39:14 A. I did.

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14:39:28 Q. Are you familiar with an
14:39:33 expert that plaintiffs have disclosed
14:39:34 in this lawsuit named Mr. Farone?
14:39:38 A. Yes.
14:39:39 Q. I believe you testified a few
14:39:41 moments ago that you knew Mr. Farone
14:39:49 when he was employed at Philip Morris?
14:39:49 A. That's correct.
14:39:49 Q. Have you had a chance to study
14:39:49 or look at his expert report in any
14:39:[illegible] way?
14:39:[illegible] A. No.
14:39:54 Q. Are you familiar with his
14:39:57 views or any testimony that he may
14:39:[illegible] provide in this case from any other
14:40:02 sources?
14:40:02 A. I have no idea what he'll
14:40:[illegible] testify about.
14:40:04 Q. You never discussed it with
14:40:09 your counsel, or you don't know what he
14:40:09 may testify to or what he has testified
14:40:09 to?
14:40:09 A. No. Oh, I know what he has
14:40:10 testified to in previous cases.
14:40:13 Q. What would that be? If you

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14:40:15 could characterize it?
14:40:19 A. Well, he has talked a lot
14:40:20 about the NOD or naturally occurring
14:40:20 denitration process that we talked
14:40:23 about earlier.
14:40:26 His views were that the
14:40:26 company should not have stopped that
14:40:28 work.
14:40:30 Q. I'm sorry?
14:40:31 A. The company should not have
14:40:35 stopped work on, quote, his NOD
14:40:38 Process.
14:40:38 And that has been the
14:40:40 substance of what I have been
14:40:43 questioned about regarding his
14:40:46 testimony in the past.
14:40:46 Q. Do you know why he left Philip
14:40:48 Morris?
14:40:49 A. I don't.
14:40:54 Q. Have you had any discussions
14:40:56 with him after he left Philip Morris?
14:41:00 At any time?
14:41:01 A. No.
14:41:02 Q. Have you ever reviewed any of
14:41:07 his work product or research, both

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14:41:07 while he was at Philip Morris or later?
14:41:10 A. I haven't reviewed work that
14:41:14 he authored. However, I was very
14:41:16 involved in the NOD project. His
14:41:23 people were working on that project.
14:41:25 So, in a sense, it was work
14:41:28 that he was responsible for but he
14:41:29 didn't do it himself.
14:41:29 Q. Do you have any reason to
14:41:32 believe or any knowledge or information
14:41:36 you've gained as to why Mr. Farone's
14:41:38 testimony wouldn't be true?
14:41:38 MR. MUEHLBERGER: Object to
14:41:39 the form.
14:41:43 A. The only thing I can tell you
14:41:46 is give you my personal views on why
14:41:49 that project was unsuccessful.
14:41:49 BY MR. HOPPER:
14:41:49 Q. The denitrification process?
14:41:54 A. Yes.
14:41:54 I don't know why he believes
14:41:57 it should have been commercialized.
14:42:00 Q. But nothing that you are aware
14:42:04 of or have knowledge of Mr. Farone that
14:42:12 would cause you to question the

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14:42:12 veracity of his testimony?
14:42:12 A. Well, I disagree with a lot of
14:42:14 what he said.
14:42:15 Q. Why is that?
14:42:18 A. Because I was responsible for
14:42:18 the Engineering Department at the time.
14:42:21 And my job was to take research that
14:42:28 was being conducted in the R & D
14:42:28 organization and try to commercialize
14:42:29 that to a useful purpose for the
14:42:33 company.
14:42:34 And I tried very hard for
14:42:34 several years, and my people tried very
14:42:38 hard for several years to scale up and
14:42:45 commercialize this technology. And it
14:42:49 just wouldn't work.
14:42:54 So, why he continues to
14:43:13 advocate that the company should have
14:43:16 commercialized this technology, I
14:43:23 really don't understand.
14:43:25 Q. Do you meet on any frequent
14:43:26 basis with Mr. Szymanczyk?
A. Sure.
Q. And do you have opportunities
to be heard about your views with

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14:43:29 respect to the responsibilities you
14:43:32 hold as a Vice-President in the
14:43:34 company?
14:43:34 A. Absolutely.
14:43:48 Q. Do you believe cigarettes are
14:43:48 addictive when they are used as
14:43:48 intended?
14:43:48 A. As I said before, I'm really
14:43:49 not qualified. I'm not a
14:43:50 pharmacologist. I'm not a physician.
14:43:54 I'm not a behavioral scientist. So, I
14:43:57 really can't answer authoritatively.
14:44:03 I'm just not qualified.
14:44:03 Q. Okay. Now, I'm asking you not
14:44:06 as an expert witness but as a fact
14:44:10 witness, which you have also been
14:44:13 rendered for in this litigation.
14:44:16 Sir, do you believe as a fact
14:44:20 witness, personally, that cigarettes
14:44:20 are addictive when used as intended?
14:44:20 MR. MUEHLBERGER: Object to
14:44:23 the form.
14:44:23 A. No. I think people enjoy
14:44:28 smoking.
14:44:28 I think it's tough for some

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14:44:29 people to quit. But I have watched a
14:44:34 lot of people quit smoking and have
14:44:34 never touched another cigarette. The
14:44:34 same is true of me.
14:44:35 So, no, I don't consider
14:44:39 cigarettes addictive.
14:44:40 Q. You say that, and yet the vast
14:44:42 number of people do have a difficult
14:44:46 time quitting; isn't that true?
14:44:46 MR. MUEHLBERGER: Object to
14:44:49 the form.
14:44:49 BY MR. HOPPER:
14:44:49 Q. Isn't it a small minority who
14:44:55 can quit and stop, and start and quit,
14:44:58 and start and stop?
14:44:58 MR. MUEHLBERGER: Object to
14:44:57 the form.
14:44:57 A. Well, I don't know. I am
14:44:57 giving you anecdotal information based
14:44:57 on my personal experience. I have
14:44:57 never really studied it extensively and
14:45:01 like I told you before, I'm really not
14:45:02 qualified to give you any more than a
14:45:05 personal point of view.
14:45:05 BY MR. HOPPER:

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14:45:08 Q. As an expert witness, you're not -- you're not holding yourself out as an expert in that regard. But you are also disclosed and provided in this litigation as a fact witness. And now I'm asking you questions as a fact witness about the addictive nature of cigarettes and your view about that.

MR. MUEHLBERGER: Object to the form. Asked and answered.

BY MR. HOPPER:

Q. And the reason I'm asking you is, you said you have access to and opportunities be heard by the President and other officers of the company or colleagues.

Have you told them that you believe cigarettes are addictive?

A. No.

Q. Have you ever shared your views -- or do you believe that smoking causes lung cancers or cancer of other organs, chronic destructive pulmonary disease and cardiovascular disease?

MR. MUEHLBERGER: Object to

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14:46:06 the form.
14:46:06 A. I think there is plenty of
14:46:06 data to suggest that if you smoke, you
14:46:06 are more likely to get certain
14:46:06 diseases.
14:46:06 BY MR. HOPPER:
14:46:06 Q. And you believe it?
14:46:08 A. I believe it.
14:46:11 Q. Now, you testified earlier you
14:46:13 were trained as a chemist, correct?
14:46:16 A. Chemical Engineer.
14:46:18 Q. But you trained in chemistry
14:46:21 extensively for that engineering job?
14:46:21 A. Extensively is a relative
14:46:25 term, but, yes.
14:46:28 Q. Well, a whole lot. You read a
14:46:30 lot of chemistry text and studied it
14:46:31 and practiced it and you understand it,
14:46:33 correct?
14:46:33 A. Yes.
14:46:33 Q. Okay, good.
14:46:35 You are familiar with the
14:46:36 term, polyaromatic hydrocarbons?
14:46:43 A. I am.
14:46:43 Q. You are familiar with

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14:46:44 benzopyrene?
14:46:46 A. I am.
14:46:58 Q. Benzo(a)pyrene?
14:46:58 A. I am.
14:46:58 Q. You are familiar with the aza-arenes?
14:46:58 A. I have heard about the classification of compounds but I have never really -- I don't know much about them.
14:47:01 Q. Such as the N-Nitrosamines, the NNKs, the NNNs?
14:47:01 A. I'm familiar with the nitrosamines.
14:47:13 Q. The tobacco-specific TSNAs?
14:47:13 A. Yes.
14:47:13 Q. Do you believe that they exist in cigarettes?
14:47:13 A. Yes.
14:47:14 Q. In the products you produce?
14:47:16 A. Yes.
14:47:16 Q. Do you believe that people who smoke your products are subjected to those highly carcinogenic arenes?
14:47:20 MR. MUEHLBERGER: Object to

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14:47:25 the form.
14:47:25 A. There are carcinogenic
14:47:28 compounds in cigarette smoke.
14:47:29 At the dose that are there, at
14:47:33 the levels that are there, I'm not
14:47:35 qualified, nor is anyone else really
14:47:37 qualified, I don't think, to make a
14:47:42 real risk assessment.
14:47:42 Q. As a fact witness, what do you
14:47:44 believe?
14:47:45 MR. MUEHLBERGER: Asked and
14:47:47 answered.
14:47:48 MR. HOPPER: No. I asked him
14:47:51 about addiction on that one, I
14:47:52 think. Not on this one.
14:47:52 A. I think there are compounds in
14:47:54 cigarette smoke that cause certain
14:47:56 types of cancer.
14:47:56 BY MR. HOPPER:
14:47:58 Q. Are you familiar with the
14:48:00 aromatic amines?
14:48:02 A. Yes.
14:48:02 Q. Aminobiphenyls?
14:48:02 A. Uh-huh.
14:48:03 Q. The naphthalenes?

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14:48:05	1	A. Yes.
14:48:09	2	Q. Do you believe those exist in
14:48:11	3	your products?
14:48:11	4	A. Yes.
14:48:12	5	Q. Are you familiar with the
14:48:14	6	aldehydes?
14:48:15	7	A. Yes.
14:48:16	8	Q. Particularly formaldehyde?
14:48:18	9	A. Yes.
14:48:19	10	Q. Do you know that that is a
14:48:20	11	suspected human carcinogen?
14:48:26	12	A. Yes.
14:48:26	13	Q. Do you believe that exists in
14:48:26	14	your product?
14:48:27	15	A. Yes.
14:48:28	16	Q. Are you familiar with the
14:48:29	17	certain organic compounds such as the
14:48:30	18	benzenes and vinyl chlorides?
14:48:30	19	A. Yes.
14:48:32	20	Q. And do you believe them to be
14:48:39	21	carcinogenic?
14:48:39	22	A. Yes.
14:48:39	23	Q. And do they exist in Philip
14:48:39	24	Morris' products?
14:48:40	25	A. Yes.

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14:48:51 Q. And are they found in mainstream or sidestream smoke from your products?

14:48:52 MR. MUEHLBERGER: Object to the form.

14:48:57 BY MR. HOPPER:

14:48:57 Q. Are they found in mainstream smoke?

14:48:57 A. In different concentrations, but yes, they are found in mainstream smoke.

14:49:00 Q. Are they found in sidestream smoke?

14:49:04 A. Yes.

14:49:10 Q. Are you familiar with certain inorganic compounds such as arsenic, chromium and polonium 210?

14:49:18 A. Yes.

14:49:18 Q. Do those exist in Philip Morris products?

14:49:19 A. I don't know.

14:49:19 Q. Why don't you know?

14:49:20 A. I just haven't seen any data on them.

14:49:23 Q. So, you are just not familiar

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14:49:27 with whether any of those inorganic
14:49:29 compounds exists?
14:49:32 A. I don't know if they are any
14:49:33 in our product or not.
14:49:46 Q. Okay. Do you believe that any
14:49:46 of these compounds can create a
14:49:49 chemical effect producing mutagenicity
14:49:57 and cariogenicity and teratogenicity?
14:49:57 MR. MUEHLBERGER: Object to
14:49:57 the form.
14:49:57 A. I'm not really qualified to
14:49:59 answer.
14:49:59 BY MR. HOPPER:
14:49:59 Q. What about as a fact witness,
14:50:01 what do you believe? Do you believe
14:50:06 they are mutagenetic, and teratogenic
14:50:06 and carcinogenic?
14:50:06 MR. MUEHLBERGER: Object to
14:50:10 the form.
14:50:10 A. I think some of the compounds
14:50:11 you mentioned are mutagenic and I think
14:50:19 some of the compounds you mentioned are
14:50:20 carcinogenic.
14:50:20 BY MR. HOPPER:
14:50:20 Q. Those that are found in Philip

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14:50:20 Morris products?
14:50:21 A. Yes.
14:50:32 Q. Do you know that Philip
14:50:33 Morris, and for that matter, other
14:50:35 defendant companies in this lawsuit
14:50:38 proved and discovered that smoking was
14:50:43 carcinogenic in various respects to
14:50:46 these different compounds and chemicals
14:50:49 as early as the late-1950s?
14:50:53 MR. MUEHLBERGER: Object to
14:50:55 the form.
14:50:59 A. No.
14:50:59 BY MR. HOPPER:
14:50:59 Q. Do you believe that was true?
14:51:01 A. I don't know. I've not heard
14:51:04 that before.
14:50:55 Q. If I showed you documents
14:50:58 showing that Philip Morris scientists
14:50:57 corresponded as early as the late-1950s
14:51:00 that they knew that; would it convince
14:51:03 you?
14:51:03 A. Well, was it a hypothesis or
14:51:05 was there data to substantiate it?
14:51:13 Q. Well, they believed there was
14:51:13 data to substantiate it.

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14:51:13 A. I don't know.
14:51:13 I would have to look at the
14:51:15 documents and try to put them in
14:51:16 context with what was going on at the
14:51:17 time to make any judgements.
14:51:17 Q. Do you believe that Philip
14:51:18 Morris, and for that matter, other
14:51:18 defendants in this lawsuit, entered
14:51:20 into an agreement with each other to
14:51:23 create a smoking controversy that
14:51:31 effectively denied the deleterious
14:51:31 health effects of smoking?
14:51:31 MR. MUEHLBERGER: Object to
14:51:33 the form.
14:51:33 A. I have never seen any evidence
14:51:34 of it.
14:51:34 BY MR. HOPPER:
14:51:34 Q. Are you aware that they hired
14:51:37 Hill and Nolton at one point in time to
14:51:42 help fabricate a health controversy?
14:51:42 MR. MUEHLBERGER: Object to
14:51:49 the form.
14:51:49 A. No, I'm not.
14:51:49 Q. You never heard anything about
14:51:49 that?

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14:51:50 A. No.
14:51:51 Q. Never discussed that with
14:51:52 anybody?
14:51:53 A. Don't even know who they are.
14:51:58 Q. Why is tobacco fine cut? What
14:52:03 does that do for your product?
14:52:09 A. I don't understand the
14:52:09 question.
14:52:09 Q. You have fine cut tobacco in
14:52:11 your products as filler?
14:52:13 A. We cut all our tobacco at
14:52:16 30-cuts per inch.
14:52:16 Q. Okay. Why is that? Why do
14:52:18 you do that?
14:52:18 A. It's -- I'm not sure I can
14:52:22 give you a reason. It has always been
14:52:29 done that way to my knowledge.
14:52:30 It's a convenient cut width
14:52:32 for packing a cigarette tube. It
14:52:35 provides enough mechanical and
14:52:39 structural stability so it doesn't come
14:52:41 out of the end of the cigarette. Has
14:52:43 reasonable burn properties, I suppose.
14:52:48 I don't know. I have never
14:52:49 really studied why 30-cuts per inch.

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14:52:49 Q. We discussed and you've
14:52:52 answered questions today about
14:52:52 flavorings.
14:52:53 What are casing?
14:52:56 A. They are similar terms.
14:53:00 Casings are generally referred
14:53:01 to when the flavor system is
14:53:05 composed of mostly humectants and
14:53:07 sometimes sugars.
14:53:09 Flavors are more typically
14:53:11 referred to as after-cut flavors. The
14:53:14 final flavor that is applied to tobacco
14:53:16 to give its signature differentiation
14:53:19 from one brand to the next.
14:53:21 Q. Do fine cut tobacco particles
14:53:27 and the use of casings and flavorings
14:53:27 make your product more inhalable?
14:53:28 MR. MUEHLBERGER: Object to
14:53:30 the form.
14:53:38 A. I don't really understand the
14:53:38 question.
14:53:38 I mean, certainly, the casings
14:53:38 and flavorings make the product
14:53:44 enjoyable to the consumer. However you
14:53:44 want to interpret that.

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14:53:48 Q. Do flavorants and additives
14:54:03 that you add, the humectants, the
14:54:03 flavorants, and I have heard some of
14:54:03 those such as -- you have used
14:54:05 chocolate even and all various types of
14:54:14 flavorants that you routinely employ --
14:54:14 do these additives increase nicotine's
14:54:17 absorption by the smoker?
14:54:20 MR. MUEHLBERGER: Object to
14:54:20 the form.
14:54:20 A. Not to my knowledge.
14:54:20 BY MR. HOPPER:
14:54:22 Q. Why do you believe that they
14:54:23 do not?
14:54:25 A. I don't have any reason to
14:54:26 believe they do.
14:54:26 Q. I understand you say you don't
14:54:33 believe they do. Why do you think they
14:54:35 do not?
14:54:35 A. I don't have any basis for
14:54:35 believing they do.
14:54:36 I can't imagine what the
14:54:41 technical argument would be.
14:54:41 Q. Why do you use high porosity
14:54:47 paper in your products to manufacture

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14:54:47 cigarettes?
14:54:58 A. To produce more ventilation.
14:54:59 Q. Do you employ low-pressure
14:55:01 drop filtration in your products?
14:55:06 A. You have to be more specific.
14:55:08 Q. Is the filters you use -- do
14:55:10 the filters that you use allow a smoker
14:55:14 to get more of a thrust from the
14:55:19 inhalation of the product?
14:55:23 MR. MUEHLBERGER: Object to
14:55:23 the form.
14:55:23 A. It's too general of a --
14:55:24 I don't understand your
14:55:25 question.
14:55:25 BY MR. HOPPER:
14:55:12 Q. Why do you believe that -- and
14:56:17 I'm asking you this in your capacity as
14:56:21 an expert witness now -- that most
14:56:23 other medications and other industrial
14:56:27 and commercial products generally
14:56:29 include, or one can find material
14:56:32 safety data sheets on those products?
14:56:35 Why has there never been any MSDA
14:56:39 sheets provided for tobacco products
14:56:41 and in particular, your products?

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A. I don't know.

MR. MUEHLBERGER: Object to the form.

BY MR. HOPPER:

Q. Do you think there should be?

14:56:43 A. Well, there are a lot of

14:56:45 health warnings on cigarettes.

14:56:47 Q. What do you mean, "a lot of

14:56:47 health warnings"?

14:56:48 A. Well, there are rotating

14:56:49 health warnings on every pack of

14:56:50 cigarettes.

14:56:53 Q. A label, you mean?

14:56:53 A. Yes.

14:56:53 Q. Since 1963?

14:56:56 A. Yes.

14:56:59 Q. And do you think that that,

14:57:01 along with the advertising and

14:57:03 promotions that you do to glamorize the

14:57:06 sale of your products, tells the whole

14:57:10 truth about all the ingredients in your

14:57:15 products?

14:57:15 MR. MUEHLBERGER: Object to

14:57:15 the form.

14:57:15 A. Well, you know, I suspect that

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14:57:16 all our consumers are not aware of all
14:57:20 the ingredients that are in our
14:57:23 cigarettes.
14:57:24 Q. Well, why did you suspect
14:57:26 that?
14:57:26 A. Because they're probably not
14:57:26 very interested in going and looking at
14:57:30 all the ingredients.
14:57:30 However, for anyone who wants
14:57:30 to have a look, they are published on
14:57:31 our website.
14:57:32 We have published ingredients
14:57:36 lists for the Federal Government for a
14:57:39 number of years.
14:57:39 It's not a secret, the
14:57:45 ingredients that we use to make
14:57:45 cigarettes. We do try to protect the
14:57:45 proprietary formulation of certain
14:57:46 brands. It is our trade secret and it
14:57:51 is what differentiates us in the
14:57:51 marketplace. But we haven't kept our
14:57:53 flavor ingredients a secret from the
14:57:58 public.
14:57:58 Q. Isn't it amazing, though, that
14:58:01 I can pick up this bottle of water

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14:58:04 right here, or any product that is on
14:58:07 the shelf as a consumer product, and it
14:58:09 will tell me what the ingredients are
14:58:12 and the percentages of the nutrients,
14:58:15 and yet, your product that is on the
14:58:17 market says absolutely nothing, zero,
14:58:20 zip, about the ingredients that are in
14:58:23 there; much less say anything about the
14:58:27 carcinogens, teratogens and mutagens;
14:58:27 isn't that true?
14:58:29 MR. MUEHLBERGER: Object to
14:58:29 the form.
14:58:32 A. It is true that there is no
14:58:32 ingredient labeling on our product.
14:58:32 BY MR. HOPPER:
14:58:35 Q. Do you think there should be?
14:58:39 MR. MUEHLBERGER: Object to
14:58:39 the form.
14:58:39 A. I don't really have a point of
14:58:40 view one way or another.
14:58:40 BY MR. HOPPER:
14:58:42 Q. As a fact witness, do you?
14:58:46 A. I don't have a point of view.
14:58:46 Q. Would it dissuade people from
14:58:46 smoking if you did publish those and

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14:58:49 put them on the side of your packages
14:58:49 the way other consumer products are
14:58:49 presented in the marketplace?
14:58:52 A. I don't think so.
14:58:52 Q. Is that because people are
14:58:54 addicted to the nicotine in them and
14:58:56 they can't --
14:58:56 MR. MUEHLBERGER: Object to
14:58:56 the form.
14:58:57 BY MR. HOPPER:
14:58:57 Q. -- no matter what they see
14:59:00 and read about it? Is that the reason?
14:59:02 A. No, that's not the reason.
14:59:04 Q. Okay.
14:59:13 Are you aware of any time
14:59:13 during the history of your company
14:59:15 since the late-'50s, of an agreement
14:59:20 that they struck between other
14:59:23 manufactures of tobacco products to
14:59:25 avoid conducting any biological
14:59:28 research on the health effects of
14:59:30 smoking?
14:59:30 MR. MUEHLBERGER: Object to
14:59:35 the form.
14:59:35 A. No.

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14:59:35 BY MR. HOPPER:
14:59:35 Q. You've never heard of that?
14:59:35 A. No.
14:59:35 Q. You don't know anything about it?
14:59:36 A. No.
14:59:36 Q. Have your conducted -- has your research and development department conducted biological
14:59:40 research on the health effects of smoking?
14:59:41 A. We have conducted biological research on our products to determine what the chemical composition of smoke is.
14:59:52 We also do certain biological assays to evaluate the relative risks of changes to our products.
14:59:59 And we have done that for quite a few years.
14:59:59 Q. Isn't it true you have also done animal research, biological research, paint studies with nitrosamines to show the effects of those in mutagenicity?
15:00:00
15:00:02
15:00:05
15:00:07
15:00:10

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15:00:10 MR. MUEHLBERGER: Object to the form.

15:00:14 A. We do inhalation studies. I've never -- I'm not aware of any mouse skin-painting studies.

15:00:17 Not to say that the company hasn't done it, but I'm just not aware.

15:00:23 BY MR. HOPPER:

15:00:24 Q. You're just not aware?

15:00:24 A. No.

15:00:26 Q. Are you aware that any biological research that was conducted at all was shipped off to Cologne, Germany, and housed in warehouses there and kept suppressed from the American public for a period of time? Have you ever heard that?

15:00:31 MR. MUEHLBERGER: Object to the form.

15:00:43 A. It's true our biological testing laboratories are in Cologne, Germany.

15:00:43 I'm not aware of any conspiracy to keep the data from the American public or anyone else.

15:00:48

15:00:49

15:00:51

15:01:01

15:01:01

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15:01:01 Q. Is it technologically
15:01:03 feasible, or has it been
15:01:03 technologically feasible, to design a
15:01:05 cigarette which would not cause an
15:01:08 increase in cancer, emphysema, asthma,
15:01:11 heart failure, cardiovascular disease,
15:01:13 or other of the signature-related
15:01:18 smoking diseases?
15:01:18 MR. MUEHLBERGER: Object to
the form.
15:01:18 A. I don't know.
15:01:20 You know, certain public
15:01:32 health authorities and regulators
15:01:32 around the world have targeted a bunch
15:01:32 of compounds that they think are
15:01:32 problematic.
15:01:32 The sad reality is, we don't
15:01:32 understand mechanistically what causes
15:01:35 these diseases.
15:01:38 So, it's very difficult to say
15:01:38 that removal of some or any of these
15:01:56 compounds would materially affect the
15:01:56 safety of the product.
15:01:56 I can tell you, however, that
15:01:56 we are working pretty hard to reduce

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15:01:56 the concentration of those targeted
15:01:56 compounds. Whether or not at the end
15:01:56 of the day it will produce a safer
15:01:56 product or not, I don't know.
15:01:56 Q. I'm curious to know why you
15:01:56 just characterized that as sadly?
15:02:01 A. Because if we understood
15:02:04 mechanistically the link between
15:02:11 certain chemicals constituents and
15:02:11 their concentrations, or combinations
15:02:11 of constituents and their
15:02:13 concentrations on disease, it would be
15:02:16 a much simpler task to modify the
15:02:20 product to reduce the risk. That's
15:02:20 why.
15:02:24 Q. You keep referring to public
15:02:25 health officials, and those health
15:02:27 officials, and the government, and
15:02:29 others; when is Philip Morris going to
15:02:32 take responsibility for admitting the
15:02:34 various constituents and the compounds
15:02:39 that are in your products that causes
15:02:43 the health effects that kill four
15:02:44 hundred and twenty-two thousand people
15:02:45 a year?

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15:02:45 MR. MUEHLBERGER: Object to
15:02:46 the form.
15:02:46 A. Well, I think the company has,
15:02:50 on a number of occasions, acknowledged
15:02:55 that there are certain substances in
15:02:58 tobacco smoke.
15:02:59 The company is not in a
15:03:05 position to know whether these
15:03:05 compounds in the concentrations that
15:03:05 they're found in cigarette smoke are
15:03:08 really what mechanistically causes the
15:03:11 disease.
15:03:11 You know, we almost have to
15:03:13 rely on what experts in the field have
15:03:16 to say.
15:03:16 BY MR. HOPPER:
15:03:17 Q. Well, let me ask you a
15:03:22 question, and you are an expert witness
15:03:22 in this litigation.
15:03:22 And I'm asking the question
15:03:25 somewhat by example, because I want to
15:03:28 know what your impression is and what
15:03:33 your opinion is as an expert.
15:03:35 There is a legal term of art
15:03:37 that lawyers use called res ipsa

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15:03:40 loquitur, which means: The thing
15:03:40 itself happened.
15:03:41 Now, if an airplane falls out
15:03:46 of the sky, there is strict liability
15:03:47 associated with that, okay?
15:03:49 And the airline industry and
15:03:51 the government and the others take
15:03:54 responsibility to address that and deal
15:03:57 with it even though they may not know
15:04:00 mechanistically what caused it to
15:04:03 happen.
15:04:04 How is it that you can
15:04:05 continue, as a company, to fall back on
15:04:09 something like not knowing the
15:04:11 mechanisms, or fully understanding the
15:04:13 constituents of your products when you
15:04:16 know full well by now and, in fact,
15:04:19 knew many, many years ago, many, many
15:04:21 years ago, back to the late-'50s, the
15:04:23 health effects of your products, and
15:04:25 your own scientists had identified
15:04:29 these effects; and yet you never
15:04:30 stepped to the forefront to do anything
15:04:34 about it. In fact, you not only didn't
15:04:34 step to the forefront, you suppressed

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15:04:35 the information and sent it off to
15:04:37 Cologne.
15:04:37 MR. MUEHLBERGER: Object to
15:04:39 the form.
15:04:39 A. Well, I don't know about
15:04:43 suppressing information.
15:04:43 All I was trying to point out
15:04:43 to you is that there are a lot of
15:04:45 substances in tobacco smoke. They are
15:04:47 there in various concentrations,
15:04:51 depending upon the brand, how the
15:04:53 cigarette is smoked. There are a lot
15:04:58 of variables.
15:04:58 We do know, the epidemiology
15:05:00 is pretty clear that if you smoke
15:05:03 cigarettes, you are more likely to get
15:05:06 certain diseases.
15:05:06 It would be -- all I'm saying
15:05:07 to you is, it would be an easier
15:05:10 problem to solve if we knew
15:05:13 specifically what compounds or what
15:05:15 combination of compounds, and what
15:05:17 concentrations caused those diseases.
15:05:20 Producing a safer product
15:05:22 would be much easier.

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15:05:24 Having said that though, we
15:05:25 are working on the whole list of the
15:05:27 compounds. And we are very actively
15:05:30 working on the whole list of compounds.
15:05:32 We are make great progress on reducing
15:05:34 the concentration of these compounds.
15:05:35 As I told you about Oasis and
15:05:37 Accord earlier today, the smoke
15:05:40 chemistry of these products and the
15:05:42 biology of these products is
15:05:46 significantly reduced from conventional
15:05:48 cigarettes.
15:05:48 But still today, we couldn't
15:05:49 make a safety claim because we really
15:05:49 don't know. The only thing we can say
15:05:51 is, look, experts have said these
15:05:54 things are harmful. We have reduced
15:05:56 the concentration by this amount. That
15:06:00 is all we can say now.
15:06:05 The epidemiology takes twenty,
15:06:05 thirty years to reap. So, there isn't
15:06:08 enough history with these products to
15:06:08 really know if they're safe or not.
15:06:09 That's all we can do. That's
15:06:09 the limit of technology today.

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15:06:10 BY MR. HOPPER:

15:06:10 Q. And yet, the overwhelming number of the health and public health community so vehemently disagree with you. Are you that far out of the dark and out of the mainstream of scientific peer-reviewed literature?

15:06:28 MR. MUEHLBERGER: Object to the form.

15:06:30 A. I don't they're disagreeing with us. I think we're listening to them and taking them at their word.

15:06:32 They are publishing papers that say substance A, B, C, D, you read off a lot of them, benzo(a)pyrene --

15:06:38 Q. Sure.

15:06:40 A. -- formalyde, you name it. You could go down the list again. We are not disputing the list.

15:06:45 We are saying that these compounds are potentially serious -- have potentially serious health consequences and we are working as hard and pulling as much technology as we can to reduce the delivery of those

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15:06:58 things to smokers.
15:06:58 Q. Okay. With that in mind, and
15:07:00 having said that then, has your company
15:07:01 ever set a standard for TSNAs?
15:07:05 A. We would like to remove them
15:07:08 all. And we are working hard to remove
15:07:11 them all. And we are making good
15:07:18 progress in reducing the concentration
15:07:18 of TSNAs.
15:07:31 Q. Is there technology available
15:07:31 to reduce the gas phases and the gas
15:07:35 phase delivery in nicotine and
15:07:38 cigarettes?
MR. MUEHLBERGER: Object to the form.
Q. You want me to say it again? Ask it again?
A. Yes, please.
BY MR. HOPPER:
15:07:40 Q. Is there technology available
15:07:42 to reduce gases in the gas phase
15:07:46 delivery of nicotine -- in the gas
15:08:02 phase delivery of cigarettes.
15:08:02 MR. MUEHLBERGER: Same
15:08:02 objection.

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15:08:02 BY MR. HOPPER:
15:08:02 Q. Is there technology available
15:08:02 to do that?
15:08:02 A. I'm not sure.
15:08:02 Q. I'm referring to what you
15:08:02 talked about and testified to the Jury
15:08:03 to earlier today, about the free
15:08:05 nicotine being suspended or available
15:08:22 in the gas phase.
15:08:22 A. In supercrictical CO2?
15:08:22 Q. Yes.
15:08:22 But not just in an
15:08:22 experimental stage. It exists in
15:08:22 cigarettes when one smokes them in the
15:08:22 gas phase.
15:08:22 A. Uh-huh.
15:08:22 Q. Is there technology available
15:08:22 to reduce that, at this time?
15:08:22 A. I'm not aware of any, although
15:08:25 most of the nicotine, almost all of the
15:08:26 nicotine is in the particulate phase.
15:08:37 Q. Do you know Scott Osborne?
15:08:40 A. I don't believe so.
15:08:47 Q. Do you know that he developed
15:08:47 a prototype at Philip Morris for an

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15:08:50 indirect cigarette that provided
15:08:50 sensory impact, as you call it, but
15:08:55 without the harmful effects of smoking?
MR. MUEHLBERGER: Object to the form.
A. I'm not familiar with the work, no.
BY MR. HOPPER:
15:08:58 Q. You don't know anything about
15:09:00 that at all?
15:09:02 A. If you have a document, I will
15:09:07 be happy to read it. But I don't
15:09:07 recall it.
15:09:09 Q. Well, I don't, your counsel
15:09:12 will be happy to know.
15:09:34 Do you believe -- as an expert
15:09:37 witness, is it your opinion that a
15:09:42 reduction of tar produces a safer
15:09:44 cigarette?
15:09:45 A. Yes.
15:09:45 Q. Why?
15:09:50 A. The less dose of those
15:09:51 chemicals, the less risk.
15:09:56 Q. Are you taking steps to reduce
15:09:56 tars in your products?

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15:09:58 A. Well, we have a whole -- produce a whole series of cigarettes with very different tar deliveries. It's a consumer choice.

15:10:06 But let me take you back to the conversation we had in reducing the concentration of certain chemical constituents --

Q. Excuse me one second.

The conversation we -- you and I had?

A. You and I had.

Q. I asked you questions and you answered them.

A. Sorry.

Q. No, that's okay. With respect to -- now go ahead.

A. Let me respond to the answer I gave you when you were asking me about certain chemical constituents in cigarettes.

Q. Okay.

A. One approach is to reduce the total amount of tar that is delivered to the consumer or that the smoker is

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15:10:38 exposed to.

15:10:39 Another approach is to alter
15:10:41 the composition of that tar, and remove
15:10:45 a lot of the constituents that we
15:10:48 talked about.

15:10:49 We are working on both. I
15:10:49 mean, we offer products with varying
15:10:51 tar deliveries. We offer products that
15:10:56 have a very different tar composition,
15:11:00 and that is the Accord product that I
15:11:05 told you about earlier.

15:11:07 Q. All right.

15:11:11 MR. HOPPER: Can we go off the
15:11:13 record for just a moment?

15:11:16 VIDEOGRAPHER: Going off the
15:11:19 record at 3:11 P.M.

15:11:54 (FOLLOWING A BRIEF RECESS, THE
15:11:54 FOLLOWING PROCEEDINGS WERE HAD:)

15:12:05 VIDEOGRAPHER: Back on the
15:12:13 record at 3:12 P.M.

15:12:13 BY MR. HOPPER:

15:12:16 Q. Mr. Burnley, I haven't asked
15:12:17 you any questions about you personally.

15:12:22 Do you have a family?

15:12:23 A. I don't.

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15:12:25 Q. Have you ever been married?
15:12:27 A. No.
15:12:28 Q. So, you don't have any kids
15:12:31 then?
15:12:32 A. No.
15:12:33 Q. Do you have brothers and
15:12:34 sisters?
15:12:34 A. I have a brother.
15:12:36 Q. Does he smoke?
15:12:37 A. Yes.
15:12:38 Q. Does he work in the tobacco
15:12:42 industry?
15:12:42 A. No.
15:12:44 Q. Do your parents smoke?
15:12:47 A. They both smoked, yes.
15:12:50 Q. Are they still alive, if I may
15:12:55 ask?
15:12:56 A. My mother is. My father is
15:12:57 not.
15:12:57 Q. Is she still smoking?
15:12:57 A. No.
15:13:06 MR. HOPPER: Counsel, I don't
15:13:07 believe I have any further
15:13:08 questions of the witness. I don't
15:13:08 know if you --

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15:13:11 MR. MUEHLBERGER: Thank you very much.

15:13:11 We have nothing.

15:13:13 VIDEOGRAPHER: Concluding the deposition at 3:13 P.M.

15:13:17 (Whereupon, the deposition was concluded and the witness was excused.)

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C E R T I F I C A T E

I, BETTY C. GODSO, CERTIFIED SHORTHAND REPORTER, #87189, REALTIME REPORTER, REGISTERED PROFESSIONAL REPORTER #005405, AND DEPUTY CLERK OF COURT IN AND FOR THE STATE OF LOUISIANA DO HEREBY CERTIFY; THAT THE WITNESS WAS SWORN TO TELL THE TRUTH, THE WHOLE TRUTH, AND NOTHING BUT THE TRUTH IN SAID CAUSE; THAT THE FOREGOING TESTIMONY WAS TAKEN BY ME IN STENOTYPE AND THEREAFTER REDUCED TO TYPEWRITING BY ME OR UNDER MY SUPERVISION; AND THE FOREGOING PAGES CONTAIN A FULL AND CORRECT TRANSCRIPTION OF ALL THE TESTIMONY TO THE BEST OF MY ABILITY AND UNDERSTANDING; THAT I AM NOT OF KIN OR ASSOCIATED WITH ANY OF THE PARTIES TO SAID CAUSE OF ACTION, OR THEIR COUNSEL, AND THAT I AM NOT INTERESTED IN THE EVENT THEREOF; THAT THE ORIGINAL WAS PROVIDED TO TAKING COUNSEL FOR THEIR CUSTODY AND CONTROL.

Betty Godso

BETTY C. GODSO, CSR #87189, RPR #005405

OFFICIAL SEAL
BETTY C GODSO
Certified Court Reporter
in and for the State of Louisiana
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October 20, 2000

James P. Muehlberger, Esq.
Shook, Hardy & Bacon
One Kansas Place
1200 Main Street
Kansas City, Missouri 64105-2118

RE: Scott v. American Tobacco, et al

Dear Mr. Muehlberger:

Reading and Signing was not waived by Mr. Harold Burnley.

Please have him them read the transcript, make any changes he feels necessary, and return the Witness Certificate along with the Errata Sheet(s).

Please keep a copy for your transcript, and please remind the deponent not to write on the transcript itself, but to make any changes on the Errata Sheet.

Upon receipt, we will forward to other counsel in this matter.

Very truly yours,

Betty C. Godso, CCR, RPR

/bcg

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INSTRUCTION SHEET FOR READ AND SIGN

THE ENCLOSED ERRATA SHEET IS FOR YOUR USE IN NOTING ANY INSTANCES YOU FEEL THE COURT REPORTER MISHEARD, MISUNDERSTOOD OR MISSPELLED WHAT YOU SAID IN YOUR DEPOSITION. IF YOU HAVE ANY QUESTIONS REGARDING A CHANGE YOU WISH TO MAKE, PLEASE CONSULT WITH YOUR ATTORNEY.

YOU SHOULD NOT BE CONCERNED WITH CHANGING MINOR SPELLINGS AND/OR PUNCTUATION UNLESS YOU FEEL IT AFFECTS THE MEANING OF YOUR TESTIMONY. IF YOU NEED TO CORRECT A SPELLING, IT IS ONLY NECESSARY TO DO IT ONCE, NOT EVERY TIME IT OCCURS IN THE TRANSCRIPT. YOU SHOULD NOT MAKE CORRECTIONS TO THE SPOKEN WORD OF ANYONE BUT YOURSELF. YOU SHOULD NOT EXPECT A TYPED, VERBATIM TRANSCRIPT TO READ SMOOTHLY AS A CAREFULLY WORDED LETTER, REPORT, OR MANUSCRIPT.

MOST RULES OF PROCEDURE REQUIRE THE SIGNED WITNESS' CERTIFICATE TO BE RETURNED WITHIN 30 DAYS. CHECK WITH YOUR ATTORNEY, OR THE ATTORNEY WHO REQUESTED YOU READ AND SIGN IF YOU HAVE QUESTIONS.

IF THERE ARE NO CHANGES, PLEASE RETURN ONLY THE SIGNED WITNESS' CERTIFICATE.

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WITNESS' CERTIFICATE

I HAVE READ OR HAVE HAD THE FOREGOING TESTIMONY READ TO ME AND HEREBY CERTIFY THAT IT IS A TRUE AND CORRECT TRANSCRIPTION OF MY TESTIMONY WITH THE EXCEPTION OF ANY ATTACHED CORRECTIONS OR CHANGES.

_____

WITNESS' SIGNATURE DATE

Harold Burnley

PLEASE INDICATE

( ) NO CORRECTIONS

( ) CORRECTIONS, ERRATA SHEET ENCLOSED

Return to attorney or: Accu-Tech Reporters; P.O.Box 45212;Baton Rouge, LA 70895

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ERRATA SHEET

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Please return the signed Witness Certificate along with any changes either to the attorney who has requested that you read and sign or to: Accu-Tech Court Reporters; P.O. Box 45212, #446; Baton Rouge, LA 70895.

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CIVIL DISTRICT COURT FOR THE PARISH OF ORLEANS

STATE OF LOUISIANA

NO. 96-8461 DIVISION "K" DOCKET NO.

GLORIA SCOTT and DEANIA M. JACKSON,
on behalf of themselves and all others similarly situated

Versus

THE AMERICAN TOBACCO COMPANY, INC., et al

FILED: _____ _____
DEPUTY CLERK

NOTICE OF VIDEO DEPOSITION OF HAROLD G. BURNLEY FOR DISCOVERY PURPOSES AND/OR FOR USE AT TRIAL

To: **Harold G. Burnley**
Through his attorney of record:
Scott E. Delacroix, Esq.
Adams & Reese
One Shell Square, Suite 4500
New Orleans, Louisiana 70139

And to: **All Defendants**
Through Liaison Counsel:

Scott E. Delacroix, Esq.
Adams & Reese
One Shell Square, Suite 4500
New Orleans, Louisiana 70139

Phillip A. Wittmann, Esq.
Stone Pigman
546 Carondelet Street
New Orleans, Louisiana 70130

Definitions and Instructions

A. **Document** - As used herein, the term "document" shall be construed broadly and expansively to include any medium upon which information is recorded, including, but not limited to, writings, records, letters, correspondence, minutes, memoranda, notes, e-mails, Internet and World Wide Web information, photocopies, facsimiles, transcripts, graphs, contracts, agreements, applications, forms, photographs, videotapes, audiotapes, microfilm, charts, studies, tables, calculations, analyses, summaries, compilations, advertisements, fliers, mailings, brochures, binders, notebooks, manuals, notices, acknowledgments, forms, and any draft of the foregoing, which is, or has been, at any time, in the control, custody or possession of the deponent, Philip Morris, Inc., Philip Morris USA, Philip Morris Worldwide Scientific Affairs, and/or parties acting on behalf of Philip Morris, Inc., Philip Morris USA, and/or Philip Morris Worldwide Scientific Affairs, including, but not limited to, the deponent, and/or other employees, officers, managers, directors, parent corporations, predecessors-in-interest, subsidiaries, associates, experts, consultants, agents, public relations advisors, advertising agents or consultants or advisors, investigators, researchers, scientists, secretaries, assistants, or attorneys.

B. **Philip Morris** - Shall refer to Philip Morris, Inc., Philip Morris USA, Philip Morris Worldwide Scientific Affairs, and/or all parties acting on behalf of Philip Morris, Inc., Philip Morris USA, and/or Philip Morris Worldwide Scientific Affairs, including, but not limited

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to, employees, officers, managers, directors, parent corporations, predecessors-in-interest, subsidiaries, associates, experts, consultants, agents, public relations advisors, advertising agents or consultants or advisors, investigators, researchers, scientists, secretaries, assistants, or attorneys.

C. Defendant - Shall refer to each Defendant, and all parties acting on behalf of that Defendant, including, but not limited to, employees, officers, managers, directors, parent corporations, predecessors-in-interest, subsidiaries, associates, experts, consultants, agents, public relations advisors, advertising agents or consultants or advisors, investigators, researchers, scientists, secretaries, assistants, or attorneys.

D. Privilege - If a privilege is claimed over any document, (as "document" is defined above), please provide: (a) the identity of the author of the document, (b) the identity of the recipient of the document, (c) the date that the document was prepared, (d) a brief description of the document, and (e) the basis upon which the privilege is claimed, including the statute, rule or decision upon which the claim of privilege is based.

E. Relates - Shall be interpreted broadly and expansively, to include relating, referring, pertaining, evidencing, supporting, describing, mentioning, including, contradicting, or compromising.

F. The terms "and" and "or" and "and/or" shall be construed broadly and expansively as "and/or", and shall not be construed to limit the documents or information sought in any manner.

G. Depositions to be used for both discovery and trial purposes. These depositions are noticed for all purposes, including trial on the merits, and are to be video and audio depositions.

H. Video and audio depositions. These depositions are noticed for all purposes, including trial on the merits, and are to be video, audio, and transcribed depositions.

I. Disputes. All disputes and objections shall be brought to the Special Master or District Court Judge in Civil District Court, New Orleans, Louisiana, for resolution and ruling, and *not* to Judges in other venues or jurisdictions.

J. Conformity with Case Management Order. The depositions herein noticed shall be in conformity with the Court's Case Management Order. *See Case Management Order No. 2, pages 6-9, Paragraph 16(c),(d).* (attached as Exhibit "A").

DEPOSITION NOTICE

PLEASE TAKE NOTICE that, commencing on Tuesday, October 10th, 2000, at 9:00 a.m. (EST), or as soon thereafter as practically possible, and continuing until concluded from day to day, at The Jefferson Hotel, located at 101 Franklin Street, in Richmond, Virginia, the Plaintiffs, Gloria Scott, Deania Jackson, and a certified class of similarly situated individuals, will take the deposition upon oral examination of Harold G. Burnley, who has been identified in defendants' Preliminary Witness List and Philip Morris Incorporated's Expert Witness List as an expert expected to testify about manufacturing and other issues, pursuant to the Louisiana Code of Civil Procedure and *Scott* CMO2, before an officer duly authorized to

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administer oaths, and by videotape, for all purposes, including perpetuation and use at trial. You are invited to attend and to participate as you deem appropriate.

DOCUMENT REQUESTS

PLEASE TAKE FURTHER NOTICE that deponent is hereby requested, pursuant to the Louisiana Code of Civil Procedure, the recommendations of the Special Master, and *Scott* CMO2, to produce and to permit the Plaintiffs to inspect and photocopy all documents set forth below:

1. A copy of the deponent's most current *curriculum vitae*;

2. Any and all documents relied upon to form the deponent's opinions in the case of *Gloria Scott, et al vs. The American Tobacco Company, et al.* and/or that deponent intends to rely upon, including, but not limited to, any and all documents identified or referenced in the deponent's expert report, and further including, but not limited to, any and all industry documents, medical records, articles, books, book chapters, or other medical or scientific publications or data, (whether published or unpublished).

3. Any and all documents referenced or identified in Reliance Documents (attached hereto as Exhibit "B").

4. Any and all deposition and/or trial testimony that deponent has given in any lawsuit, including written transcripts and/or videotapes, and any and all testimony that deponent has given before or submitted to any legislative or regulatory body, committee, sub-committee, or agency, including written transcripts and/or videotapes and/or affidavits, relating to smoking and health issues, personal injuries arising from tobacco use, cigarette advertising or marketing, lung cancer, throat cancer, bladder cancer, chronic obstructive pulmonary disease, heart disease, addiction, medical monitoring, and/or smoking cessation, including, but not limited to, the testimony identified in Philip Morris' Answer to Interrogatory No. 1, (i.e. *Barnes v. American Tobacco Company*, No. 96CV-5903, USDC E.D.Pa., Sept. 16-17, 1997, *Engle v. R.J. Reynolds*, No. 94-08273 CA (20) Dade County Circuit Court, May 28, 1998, *Williams v. Philip Morris*, No. 9705-03957, Multnomah County Circuit Court, March 15, 1999, *Iron Workers Local 17 (OH) v. Philip Morris*, No. 1:97CV 1422, USDC N.D.Ohio, March 9, 1999, *Philip Morris v. ABC*, No. 760CL94X00816-00, City of Richmond Circuit Court, May 31 - June 2, 1995), which has not yet been produced to *Scott* plaintiffs' counsel.

5. Any and all complete original file(s) pertaining to *Scott v. American Tobacco, et al.* including, but not limited to, any and all expert billing records, consultation agreements, and/or employment agreements of any nature between deponent and Philip Morris.

6. Any and all documents that are not reflected in deponent's *curriculum vitae* or expert report, but which are related to or arise from any research or other scientific endeavor conducted by the deponent relating to smoking and health issues, smoking cessation, medical monitoring, routine medical tests or screening, cigarette advertising or marketing, and cigarette or tobacco research of any kind.

7. Any and all documents provided to deponent by counsel for Philip Morris and/or counsel for any other defendant and/or any other expert hired by Philip Morris or any other defendant, in *Scott*, and/or in any other litigation relating to smoking and health issues, personal injuries arising from tobacco use, cigarette advertising or marketing, medical monitoring, and/or smoking cessation.

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8 Any and all documents pertaining to Plaintiffs, Philip Morris, other defendants, and/or any other matter which is relevant to *Scott*, obtained from any source.

9 Any and all notes pertaining to Ms. Scott or Ms. Jackson.

10. Any and all exhibits, compilations, summaries, notes, memoranda, audiotapes, videotapes, and/or PowerPoint or other computer and/or slide presentations, prepared by or at the request of deponent relating or in any way pertaining to *Scott*.

11. Any and all unprivileged correspondence, including electronic mail, between deponent and counsel for Philip Morris and/or any other expert hired by any defendant in the *Scott* litigation. (If you contend that any communications are privileged, please provide information per instruction D, *supra*.)

12. Copies of any and all public statements, including those made for the media or legislative or regulatory purposes, relating to the etiology of chronic diseases, smoking cessation, routine tests or screening, medical monitoring, cigarette advertising or marketing, cigarette design, tobacco additives and/or constituents, and/or addiction.

13. Copies of any and all lecture notes, outlines, PowerPoint or other computer and/or slide presentations, and/or course materials, for any and all lectures and/or seminars prepared or presented in any teaching or educational capacity, relating to the etiology of chronic diseases, smoking cessation, routine tests or screening, medical monitoring, cigarette advertising or marketing, cigarette design, and/or addiction.

14. Any and all documents, including, but not limited to, copies of correspondence exchanged with persons or organizations, relating to the etiology of chronic diseases, smoking cessation, routine tests or screening, medical monitoring, cigarette advertising or marketing, cigarette design, and/or addiction.

15. Any and all summaries, evaluations, memoranda or reports prepared by deponent that relate to his opinions and/or anticipated testimony in *Scott*.

16 Any and all articles, books, or book chapters written by or contributed to by deponent that are in press or otherwise not publicly available.

17. Any documents, including electronic communications and/or notes, pertaining to the attachments in deponent's expert report.

18. Any and all documents relating to projects, research, budgets, and/or cost studies, relating to medical monitoring and/or smoking cessation devices, protocols, treatments, and/or programs.

19. Any and all documents relating to animal studies.

20 Any and all documents evidencing or otherwise relating to the presence or addition of ammonia, ammonia compounds, and/or acetehaldyde, in or to Philip Morris cigarettes.

21. Any and all documents evidencing or otherwise relating to the addition of nicotine compounds, nicotine analogues, nicotine blends, and/or nicotine, to Philip Morris cigarettes.

22. Any and all documents relating to the development, research, marketing, and/or promotion of any proposed or actual "safe", "safer", "healthy", "healthier", and/or "denicotinized"

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cigarettes or other nicotine products.

23. Any and all documents relating to the cigarette manufacturing process and "other issues" as reflected in paragraph 10, page 2, of Defendants' Witness List.

Respectfully submitted,

RUSS M. HERMAN, Bar No. 6819
STEVE HERMAN, Bar No. 23129
Herman Herman Katz & Cotlar, LLP
820 O'Keefe Avenue
New Orleans, Louisiana 70113
Telephone: (504) 581-4892
Fax No. (504) 561-6024

CERTIFICATE OF SERVICE

I hereby certify that the above and foregoing Notice has been served upon deponent, who is a current Philip Morris employee and/or has been listed as a fact and/or expert witness by Philip Morris, through defendant's counsel of record, Scott Delacroix, by hand, and upon Defense Liaison Counsel and the Special Master, by placing a copy of same in the U.S. Mail, properly addressed and postage pre-paid, this 15th day of September, 2000.

RUSS M. HERMAN

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CIVIL DISTRICT COURT FOR THE PARISH OF ORLEANS

STATE OF LOUISIANA

NO. 96-2461 DIVISION "K"

GLORIA SCOTT, ET AL

VERSUS

THE AMERICAN TOBACCO COMPANY, INC., ET AL

CASE MANAGEMENT ORDER NO. 2 ("CMO 2")

This order governs these proceedings and remains in effect up to the signing of a superceding Case Management Order or the Pre-Trial Order by this Court. This order is final and no modifications to this order are permitted without written motion to the Court, and upon a showing of good cause.

1. Provisions Continued from Case Management Order No. 1

Paragraphs 17, 20, 21, 23, 24, 25, 26, 27, 28 and 30 of Case Management Order No. 1 shall remain in force and effect.

2. Dates

Any reference here to "days" shall refer to calendar days.

3. Stay of Discovery

The stay of discovery imposed in Paragraph 15 of Case Management Order No. 1 is hereby lifted. All discovery shall be conducted in strict accord with the provisions of this Order.

4. Rules Governing Proceedings

Except as otherwise provided in this Court's prior orders, in this order, or by further order of this court, the Louisiana Code of Civil Procedure and the Uniform Rules of the Civil District Court for the Parish of Orleans shall govern all further proceedings herein.

5. Transcripts

All hearings and status conferences in this matter, including those before the Special Master, shall be transcribed in the absence of agreement to the contrary by all parties. The cost of such transcription shall be divided equally between plaintiffs and

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defendants.

6. Substantive Issues of Law

Nothing set forth in this Case Management Order shall be deemed to effect any substantive right, claim, or defense of any party to this matter or to constitute any ruling or order on any question of law.

7. Admission of Counsel

Any non-Louisiana attorney who wishes to appear *Pro Hac Vice* in this litigation and who has not previously moved for and obtained an order of admission can file and serve a Notice of Appearance Pro Hac Vice. The Notice shall indicate the Bars of all the States in which the attorney is a member in good standing. Upon filing and service of the Notice, the attorney shall be deemed admitted Pro Hac Vice. No further court order shall be required for such admission. Any non-Louisiana attorneys so admitted shall be subject to and governed by the Louisiana Rules of Professional Conduct.

8. Amendment of Pleadings

All amendments to pleadings related to the Phase I, II and III trials shall be filed on or before August 1, 2000. Defendants shall have thirty (30) days after service of such pleadings to file responsive pleadings.

9. Pending Exceptions and Motions

There currently are pending the following exceptions and motions:

a. Declinatory Exception of Lack of Personal Jurisdiction of UST Inc.

b. Declinatory Exception of Lack of Personal Jurisdiction of The Tobacco Institute, Inc.

c. Exception of Lack of Personal Jurisdiction of BATUS Holdings, Inc. and BATUS, Inc.

d. Declinatory Exception of Lack of Personal Jurisdiction of American Brands, Inc.

e. Declinatory Exception of Lack of Personal Jurisdiction of RJR Nabisco, Inc.

f. Exception of Lack of Personal Jurisdiction of Loews Corporation

g. Exceptions Incorporated in the Answer filed by R.J. Reynolds Tobacco

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Company

h. Exceptions incorporated in the answer filed by Philip Morris Companies, Inc.

i. Exceptions incorporated in the answer filed by Brown and Williamson Tobacco Corporation, individually and as successor by merger to the American Tobacco Company

j. Motion of Brown and Williamson Tobacco Corporation, individually, and as successor by merger to The American Tobacco Company to strike Exhibit S.

As set forth in Paragraph 21 of Case Management Order No. 1, the failure of any defendant to fix such declinatory and/or dilatory exceptions or other motions for hearing, and the participation by such defendants in other proceedings in this case, shall not constitute the waiver of any defendants' exceptions and shall be without prejudice to, and with full reservation of, all rights and remedies of such defendants relating to their jurisdictional and other exceptions. Notwithstanding the foregoing, any defendant may fix its exceptions and/or motions for hearing on any of the hearing dates identified in any Order of this Court, or subsequently identified by further order of this Court, as a hearing date for this matter, conditioned upon compliance with the filing and notice requirements set forth in this Case Management Order.

10. Filing of Motions and/or Fixing Pending Matters for Hearing

a. Scheduling Pending Matters for Hearing

Any defendant may fix its exceptions and/or motions for hearing on any of the dates identified by this Court as a hearing date in this matter. In order to fix a pending exception and/or motion for hearing, such defendant must notify plaintiffs of its intent to bring forth the exceptions and/or motion for hearing at least twenty-one (21) days prior to the scheduled hearing date. Any opposition to the exception and/or motion must be filed with the Court at least seven (7) days in advance of the hearing date. Any reply memorandum must be limited to the matters raised in the opposition and must be filed with the Court at least two (2) days in advance of the hearing date. No further briefing is

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permitted.

b. Filing and Scheduling New Matters for Hearing

Except as otherwise set forth in this Order or a Pre-Trial Order, any party may file a motion and schedule it for hearing on one of the dates identified by this Court as a hearing date for this case, or subsequently identified by further order of this Court as a date for this matter. Except as otherwise set forth in this Case Management Order, such motion must be filed and scheduled for hearing at least twenty-one (21) days prior to the scheduled hearing date. Any opposition to the motion must be filed with the Court at least seven (7) days in advance of the hearing date. Any reply memorandum must be limited to matters raised in the opposition and must be filed with the Court at least two (2) days in advance of the hearing date. No further briefing is permitted excepted by leave of court for good cause shown.

All motions and memorandum must be served upon the Special Master at the time of service on the adverse party.

c. Dispositive Motions

All dispositive motions in this matter shall be filed in accordance with the deadlines set forth in this CMO. All such motions must be served on the Special Master at the time of service on the adverse parties.

11. Discovery Disputes

All discovery disputes are hereby referred to the Special Master for expedited hearing and resolution.

12. Discovery Requests and Responses

Pursuant to Louisiana Code of Civil Procedure Article 1474 and Local Rule 9, Section 3, discovery requests and responses shall not be filed with the Court except that they may be appended to a motion or opposition to which they are relevant or as otherwise authorized by the Louisiana Code of Civil Procedure.

13. Service of Discovery on the Special Master

Copies of all written discovery material shall be served on the Special Master.

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14. Service

A courtesy copy of all pleadings, motions, briefs, or other documents filed or otherwise presented to the Court or the Special Master by plaintiffs shall be delivered by hand or by fax to defendants' liaison counsel and to local counsel for Brown &Williamson Tobacco Corporation, Lorillard Tobacco Company, The Tobacco Institute and United States Tobacco Company no later than 4:00 p.m. on the earliest date the pleading, motion, brief or other document is filed and presented. If the pleading, motion, brief or other document is to be submitted to the Court for action on the same day as filing or presentation, it must be delivered by hand or by fax before presentation to the Court.

Five courtesy copies of all pleadings, motions, briefs or other documents filed or otherwise presented to the Court or the Special Master by any defendant shall be delivered by hand or by fax to plaintiffs' liaison counsel no later than 4:00 p.m. on the earliest day the pleading, motion, brief or other document has been filed or presented. If the pleading, motion, brief or other document is to be submitted to the Court for action on the same day as filing for presentation, it must be delivered by hand or by fax, before presentation to the Court.

15. Class Notice and Notification Plan

Notice to the Class shall occur in accordance with the orders of this Court.

16. Phase I, II and III Trial Discovery

The stay of discovery is hereby lifted. Accordingly, discovery relating to the named plaintiffs' individual claims, class-wide claims and all the issues to be adjudicated in the Phase I, II and III trials, as set forth in this Court's Trial Order 1, including records collection, written discovery and depositions, shall be conducted according to the following schedule, which may be extended or modified only for good cause shown, upon contradictory hearing.

a.) Records Collection

If not previously provided, within twenty-one (21) days of entry of the CMO, named plaintiffs and class representatives shall execute and provide to Defendants' Liaison Counsel authorizations for the release of medical, prescription, psychiatric, employment, insurance, union and educational records and a verified list of all of their respective

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treating physicians and of all medical facilities where they have received treatment, and shall cooperate so as to enable defendant to obtain such records expeditiously. One copy of all such medical, prescription, psychiatric, employment, insurance, union and educational records obtained by defendant shall be provided to Plaintiffs' Liaison Counsel within ten (10) days of receipt of such records by defendant.

b.) Written Discovery

i. All written discovery (Interrogatories, Requests for Production and Requests for Admission) shall be served on the opposing party on any issue pertaining to the Phase I, II and III trials no later than forty-five (45) days from the date of entry of CMO2.

ii. Within thirty (30) days after the service of written discovery, all parties are to serve any objections and responses to written discovery. All answers shall be verified in accordance with the provisions of the Louisiana Code of Civil Procedure.

iii. Any motions relating to written discovery shall be filed in accordance with the provisions of CMO2 and shall be scheduled for hearing before the Special Master.

c.) Depositions of Fact Witnesses

i. Depositions of fact witnesses, including class representatives, may be noticed immediately after the entry of CMO2. The parties shall confer with each other, third-party deponents and the Special Master (when feasible and appropriate) and attempt to agree on the scheduling of depositions as to time and place. Any party noticing a deposition shall give at least ten (10) days notice of the taking of the deposition unless a shorter period of time is agreed to by the parties or ordered by the Court or the Special Master for good cause shown.

ii. All depositions may be videotaped. Any party intending to videotape a deposition shall so state in a Notice of Deposition.

iii. *Attendance and Examination:* Unless otherwise agreed to by the parties or ordered by the Court or the Special Master, depositions

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may be attended only by the parties, the parties' experts and consultants, the deponents, the deponents' attorneys, attorneys representing any of the parties in this action, in-house counsel for the parties, the employees of such counsel, and court reporters. Plaintiffs and defendants, collectively, shall designate a lead counsel in conducting the examination of the deponent, but counsel for other parties may be permitted to conduct examination on non-duplicative matters.

iv. *Conduct at Depositions:* Unless otherwise agreed by the parties, and noted on the record, the following stipulations shall apply to all depositions in this action:

1.) Unless otherwise specified by any defendant, an objection by a single defendant shall be deemed an objection by all defendants. However, unless otherwise specified, an instruction not to answer by one defendant should not be deemed an instruction not to answer by all defendants.

2.) All objections are reserved until trial or other use of the deposition, except those objections regarding the form of the question or the existence of a privilege.

3.) Counsel shall refrain from engaging in colloquy during depositions. The phrase "objection as to form" or similar language shall be sufficient to preserve all objections as to form until the deposition is sought to be used. If requested, the objecting party shall provide a sufficient explanation for the objection to allow the deposing party to rephrase the question.

4.) The counsel shall not direct or request that a witness refuse to answer a question, unless that counsel has objected to the question on the ground that the

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question seeks privileged information, information that the Court has ordered may not be discovered, or a deponent seeks to present a motion to the Court for termination of the deposition on the grounds that it is being conducted in bad faith or in such a manner as unreasonably to annoy, embarrass, or press the party or the deponent. When a privilege is claimed, the witness shall nevertheless answer questions relevant to the existence, extent or waiver of the privilege, such as the date of a communication, who made the statement, to whom and in who's presence the statement was made, other persons to whom the contents of the statement was made, any other person to whom the contents of the statement has been disclosed, and the general subject matter of the communication.

5.) Counsel shall not make objections or statements which might suggest an answer to a witness.

d.) Use of Videotape Depositions

Parties should arrange to videotape the depositions of witnesses not expected to testify live at trial. Deposition designations presented in lieu of live testimony should be presented via edited video deposition presentations incorporating the Judge's rulings on objections, the offering party's designated testimony and the opposing parties counter-designations. In the event a video deposition is not available, parties may read selected deposition designations into the record.

17. Rulings; Immediate Presentation

Disputes arising during depositions that cannot be resolved by agreement and that, if not immediately resolved, will significantly disrupt the discovery schedule or require rescheduling of the deposition, shall be presented by telephone or in person to the Special Master. In the event the Special Master is not available, the deposition shall

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continue with the issue of contention reserved until the Special Master is available. The presentation of the issue and the Special Master's ruling shall be recorded as part of the deposition.

18. Use of *Castano* Depositions

All depositions (including depositions of class representatives and fact and expert witnesses) and the entire discovery and testimonial record taken or developed in *Castano*, prior to the certification of the class on February 17, 1995, may be used in this action for any purpose as if the depositions had been taken and the record had been generated in this case.

19. Requirement to Confer

Counsel must confer at least seven (7) days prior to the filing of any motion to compel or other motion related to discovery. No party shall file a motion to compel or other motion relating to discovery unless certification is made in the record by counsel for the moving party that the parties conferred and a good faith attempt was made to resolve disputes arising from the responses to discovery.

20. Document Identification

Counsel shall develop and use a system for identifying, by unique number or symbol, each document produced or referred to during the course of this litigation.

21. Document Production

Within sixty (60) days after the entry of CMO2, the plaintiffs shall furnish the defendants with a preliminary list of documents which they believe to be relevant to the issues of the Phase I, II and III trials and a preliminary identification of documents they may introduce at each of the Phase trials, identifying the Phase trial at which the document may be offered. This list of documents shall contain the following categories of information (if said information is available):

a. The defendant the document relates to;

b. Any identifying or bates numbers contained on said document;

c. The date of the document;

d. The author of the document;

e. The recipient of any document;

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f. A brief reference description of the contents of the document;

g. An identification of the source of the document;

h. Any other identifying numbers affixed to said document;

Within thirty (30) days after the receipt thereof, the defendants shall specify which documents defendants are unable to identify from plaintiffs' list and request copies of said documents from the plaintiffs. Plaintiffs shall furnish copies of such documents to the defendants on or before ten (10) days after receipt of such request.

Within sixty (60) days after the entry of CMO2, the defendants shall also furnish the plaintiffs with a preliminary list of documents which they believe to be relevant to the issues of the Phase I, II and III trials and a preliminary identification of documents they may introduce at each of the Phase trials identifying the Phase trial at which the document may be offered. This list of documents shall contain the following categories of information (if said information is available):

a. The defendant the document relates to;

b. Any identifying or bates numbers contained on said document;

c. The date of the document;

d. The author of the document;

e. The recipient of any document;

f. A brief reference description of the contents of the document;

g. An identification of the source of the document;

h. Any other identifying numbers affixed to said document.

Within thirty (30) days after receipt of the defendants' list of documents, the plaintiffs shall specify which documents plaintiffs are unable to identify from defendants' list and request copies of said documents from defendants. Defendants shall furnish copies of such documents to the plaintiffs on or before ten (10) days after receipt of such request.

All parties shall make a continuing good faith effort to identify additional documents which may be used at trial and to supplement their listing of documents on a timely basis. All documents shown on any supplemental listings shall be subject to the time limitations for identification and assertion of privilege set forth hereinabove.

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22. Privilege Logs

Within thirty (30) days after production of the lists of documents, the parties shall identify all documents which the parties assert are protected by any privilege and shall submit a privilege log to the Special Master and serve notice to the parties of any claim of privilege in the following manner:

a. If any documents are withheld from production on the grounds of privilege or work product, the withholding party shall serve on the party requesting production, as part of the objection to production, a privilege log identifying each document withheld and specifying for each document its nature or type, its identification number, its date, its author(s), its addressee(s), the recipient(s) of copies, it's subject matter and the basis for the assertion of privilege or work product.

b. In lieu of preparing the privilege log required by subsection (a) above, the parties may, as to any set of documents which are both numerous and raise common privilege or work product issues, identify the documents for which a claim of privilege or work product is being made in a generic matter by category of document rather than on a document by document basis, provided that the description of each category is sufficient to permit the claim of privilege or work product to be evaluated by the opposing party, the Special Master and the Court.

c. The privilege log shall also be submitted as part of any responses or objections to interrogatories or document requests in which a privilege is asserted.

All motions arising from disputes related to the discovery of documents and privilege issues are hereby referred to the Special Master for expedited hearing and prompt recommendations to the Court for action.

23. Completion of Merits Discovery of Fact Witnesses

All merits discovery of fact witnesses pertaining to issues in the Phase I, II and III trials shall be completed sixty (60) days before the commencement of the Phase I trial.

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24. Expert Witness Identification and Discovery

a. Within ninety (90) days from the entry of this CMO, plaintiffs shall serve on defendants a list of expert witnesses they may call at each of the Phase trials, identifying the Phase trial at which the expert is expected to testify. Fifteen (15) days thereafter, the plaintiffs shall serve on the defendants a written report prepared and signed by the witness, which report shall include:

i.) A complete statement of all opinions to be expressed and the basis and reasons therefore;

ii.) The data or other information considered by the witness in forming the opinions;

iii.) Any exhibits to be used as a summary of or in support of the opinions;

iv.) The qualifications of the witness, including list of all publications authored by the witness within the preceding ten (10) years;

v.) The compensation to be paid for the study and testimony;

vi.) A listing of any other cases in which the witness has testified as an expert at trial or by deposition within the preceding four (4) years.

b. Within one hundred twenty (120) days from the entry of this CMO, defendants shall serve on plaintiffs a list of expert witnesses they may call at each of the Phase trials, identifying the Phase trial at which the expert is expected to testify. Fifteen (15) days thereafter, the defendants shall serve on the plaintiffs a written report prepared and signed by the witness, which report shall include:

i.) A complete statement of all opinions to be expressed and the basis and reasons therefore;

ii.) The data or other information considered by the witness in forming the opinions;

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iii.) Any exhibits to be used as a summary of or in support of the opinions;

iv.) The qualifications of the witness, including list of all publications authored by the witness within the preceding ten (10) years;

v.) The compensation to be paid for the study and testimony;

vi.) A listing of any other cases in which the witness has testified as an expert at trial or by deposition within the preceding four (4) years.

Failure to comply with the report requirements may result in exclusion of the expert's testimony at trial.

c. Depositions of expert witnesses may be noticed ten (10) days after the exchange of reports is completed and shall be completed at least seventy-five (75) days before the date of the commencement of the Phase I trial. All depositions of expert witnesses shall be conducted in accordance with the provisions set forth in Paragraphs 16(b) and 17 above.

25. Discovery of Absent Class Members

The time period for discovery of absent class members will be set by the Court after it determines the identities of those individuals who will pursue their claims against defendants in this suit.

26. Procedural and Other Motions

The Court has established a regular monthly motion day to hear motions in Trial Order 1.

27. Dispositive Motions

Any dispositive motions, including summary judgment motions, must be filed no later than ten (10) days after the close of expert discovery. All such motions shall be heard on December 6, 2000.

28. *Daubert/Foret* Motions and Motions in Limine

All *Daubert/Foret* motions and motions in limine must be filed no later than ten (10) days after the close of expert discovery. All such motions shall be heard on

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29. Preliminary and Final Witness and Exhibit Lists

a. Plaintiffs shall file and serve a preliminary list of witnesses sixty (60) days after entry of CMO2 and a final list of witnesses and a final exhibit list no later than one hundred twenty (120) days before the commencement of Phase I trial.

b. Defendants shall file and serve a preliminary list of witnesses ninety (90) days after entry of CMO2 and a final list of witnesses and a final exhibit list no later than one-hundred twenty (120) days before the commencement of the Phase I trial.

c. Presentation of Testimonial, Demonstrative and Real Evidence: Upon submission of final pre-trial exhibit lists, parties shall submit to the Court DC-ROM's containing images of the listed exhibits in standard Group IV TIFF format.

In an effort to speed the admission and display of evidence, the parties shall share a single multimedia presentation network consisting of a series of monitors and projection screens throughout the courtroom for the presentation of documents, demonstrative aids and video during the trial. This network shall be for computer signal and video signal display only and shall not require the parties to share a computer or data files.

30. Completion of Discovery

All discovery must be completed seventy-five (75) days before the commencement of the Phase I trial.

31. Verdict Forms, Jury Interrogatories and Jury Instructions

All parties shall submit to the Court and Special Master and serve upon liaison counsel proposed verdict forms, special jury interrogatories and proposed jury instructions for Phase I, II and III trials no later than seventy-five (75) days before the commencement of the Phase I trial. Objections to all such submissions shall be made in writing no later than sixty (60) days before the commencement of the Phase I trial.

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32. Written Objections to Exhibits

All objections to the admissibility of all exhibits to be offered shall be filed no later than December 8, 2000.

33. Pre-Trial Order

The final Pre-trial Order shall be signed by all parties, filed with the Court and served on the Special Master no later than December 8, 2000.

34. Pre-Trial Conference

The final Pre-Trial Conference is set for December 13, 2000 at 10:00 a.m.

35. Conferring with the Special Master

Any party may, upon reasonable notice, request the Special Master to meet and confer with the parties on any matter covered by this order. Ex parte conferences with the Special Master are prohibited.

New Orleans, Louisiana, this 24th day of February, 2000.

Richard J. Ganucheau
JUDGE, Division "K"

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CIVIL DISTRICT COURT FOR THE PARISH OF ORLEANS

STATE OF LOUISIANA

NO. 95-2451 DIVISION "K"

GLORIA SCOTT, ET AL

VERSUS

THE AMERICAN TOBACCO COMPANY, INC., ET AL

JUDGMENT

These matters came up for hearing on the 20th day of December, 1999:

1. Plaintiffs' Motion for Reconsideration of the Court's September 14, 1999 Order Requiring Television and Additional Print Notice;
2. Plaintiffs' Objections to Defendants' Notice Plan and Form of Notice;
3. Plaintiffs' Objections to Defendants' Proposed Trial Plan and Trial Issues;
4. Defendants' Objections to Plaintiffs' Notice Plan and Form of Notice;
5. Defendants' Motion for Notice Plan and Form of Notice; and
6. Defendants' Objections to the Plaintiffs' Proposed Trial Plan and Trial Issues.

After considering the Memoranda, applicable law and argument of counsel:

IT IS ORDERED that the Plaintiffs' Motion for Reconsideration of the Court's September 14, 1999 Order Requiring Television and Additional Print Notice, the Plaintiffs' Objections to Defendants' Notice Plan and Form of Notice, the Defendants' Objections to Plaintiffs' Notice Plan and Form of Notice, and the Defendants' Motion for Notice and Plan and Form of Notice are GRANTED in part and DENIED in part. The Court adopts its September 14, 1999 Order with the following modifications:

a) The print notices published in the daily and weekly newspapers and in the publications referenced in the original Order, shall be ⅓ page in size;

b) Standard fonts and font sizes shall be used in the print notice;

c) Color fonts and color borders are not to be used;

d) A standard 1 point black border shall be used to distinguish the print notice;

e) No coupons are permitted in the print notice;

f) The print notice shall be published in Sunday editions of the listed newspapers

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two times over a 30-day period;

g) The print notice shall be published in daily editions of the newspapers listed four times over a 30-day period;

h) The print notice shall be published one time in the weekly publications listed;

i) The print notice shall be published one time in the magazine publications listed;

j) The first print notice shall be published no later than sixty (60) days after the entry of this Judgment in the newspapers and the magazines listed in the Order of September 14, 1999;

k) The print notice shall contain a toll-free number which will provide tape recorded information in response to inquiries from class members; such information shall include the class definition, opt-out rights and the opt-out period, the name and telephone number of the Special Master and the address to which class members send their opt-out notices;

l) The print notice shall reference a World Wide Website address, which shall contain the print notice, the name and telephone number of the Special Master, the toll-free telephone number, opt-out rights and the opt-out period and the address to which class members send their opt-out notices;

m) The opt-out period is forty-five (45) days, such period to begin from the scheduled publication date of the last print notice in any publication. Opt-outs are to be instructed to write to the address listed in the notice or the *Scott v. American Tobacco* Special Master, Dominic J. Gianna, , 201 St. Charles Avenue, 31st Floor, New Orleans, LA 70170-3100;

n) The print notice shall be published in English and Spanish in any Hispanic publication. All inquiries in the Spanish language shall be referred to the Special Master, who will refer the inquiries to Mr. Raul Bencomo for translation;

o) The broadcast television notices ordered by this Court's September 14, 1999 Order are to be broadcast in the "average prime times" (Option 2) referenced in the October 28, 1998 Affidavit of Peter Hillsbee and the broadcast of such notice shall begin no later than sixty (60) days after entry of this Judgment. The notice must contain a brief narrative of the class definition, the toll free

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number, the World Wide Website address, the name and telephone number of the Special Master, a brief description of the class member opt-out rights and the opt-out date;

p) All notices must make clear that this action seeks to establish a fund for medical monitoring/cessation of smoking, that damages for personal injury will not be awarded, that the purpose of medical monitoring is to detect latent disease or injury and not treat it, that the class representatives are not seeking direct monetary sums, and must not suggest that class members are either entitled to monetary damages or that they will receive money for medical monitoring or cessation of smoking programs;

q) All notices must inform class members that the relief sought is a medical monitoring program which will require class members to undergo tests over some period of time and cessation of smoking programs which may require participation in medically supervised programs designed to stop them from smoking;

r) All notices must inform class members that the defendants deny all allegations of wrongdoing and liability for all damages, and claim that participation in this class action may bar monetary claims for current personal injuries;

s) All notices shall direct inquiries to the *Scott v. American Tobacco* Special Master, Dominic J. Gianna, Middleberg, Riddle & Gianna, at (504) 207-7307, 201 St. Charles Avenue, 31st Floor, New Orleans, Louisiana 70170-3100;

t) All notices must inform class members not to call the Court or the Clerk of Court;

u) Registration forms are not required at this time;

v) The content and form of the print notice, the press releases, the courthouse communications, the television notice, the internet communication and the tape-recorded information available on the toll-free number must be submitted to the Special Master and the Court at least twenty-one (21) days before publication/dissemination.

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IT IS FURTHER ORDERED THAT Plaintiffs Objections to Defendants' Proposed Trial Plan and Trial Issues and Defendants' Objections to Plaintiffs' Proposed Trial Plan and Trial Issues, are DENIED in part and GRANTED in part. The Court issues Trial Order No. 1 and Case Management Order No. 2, attached, as the Trial and Pre-Trial Plans that will govern the proceedings in this matter.

JUDGMENT READ, RENDERED AND SIGNED in New Orleans, Louisiana, this 24th day of February, 2000.

Richard J. Garbcheau
JUDGE, Division "K"

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CIVIL DISTRICT COURT FOR THE PARISH OF ORLEANS

STATE OF LOUISIANA

NO. 96-8461 DIVISION "K"

GLORIA SCOTT, ET AL

VERSUS

THE AMERICAN TOBACCO COMPANY, INC., ET AL

TRIAL ORDER 1

On September 14, 1999, this Court ordered the parties to submit to the Court and to the Special Master their proposed Trial Plans and objections to those Trial Plans. On December 20, 1999, the Court heard oral argument on this matter.

After considering the Trial Plans proposed by the parties, the objections to those Plans, the Memoranda submitted by the parties, the argument of counsel, and the Louisiana Code of Civil Procedure Article 593.1 (1984), the Court concludes that this matter shall be managed in accordance with La. C.C.P. Art. 593.1 (C)(1984). The trial of this matter shall be conducted in phases. Consequently,

IT IS ORDERED that the Phase I trial of this matter commence on the 15th day of January, 2001. The Phase I trial shall adjudicate the liability of the defendants for all damages common to the class certified by the Court on April 16, 1997. The issues in the Phase I trial relating to liability for all damages will be fault and causation, namely, whether the defendants are legally liable for damages that are alleged to be common to the class. The issues include, but are not necessarily limited to, (1) whether the class representatives smoked; (2) whether nicotine in the defendants' products caused the class representatives to smoke; (3) whether the defendants' products were defective in relation to nicotine and addiction as a matter of Louisiana law; and (4) whether the addiction of the class representatives to nicotine caused conditions or effects that justify medical monitoring or other class-wide remedies or damages, such as smoking cessation remedies, for which the defendants are legally responsible.

The Phase II trial, before the same jury, shall commence 14 days after the completion of the Phase I trial and shall, pursuant to La. C.C.P. Art. 593.1 (C)(2) determine any items of damage common to the class and the basis for the assessment of those damages, e.g., whether the class is entitled to compensation for medical monitoring

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and/or smoking cessation, and, if so, the bases for assessing those damages on a class-wide basis.

The Phase III trial, before the same jury, shall commence 14 days after the completion of the Phase II trial, and shall, pursuant to La. C.C.P. Art. 593.1 (C)(3), assess the common damages determined in the Phase II trial. On the basis of the criteria or standards established by the Phase II trial for assessing common or class damages to the members of the class, the Phase III trial will assess, or award, the common damages. The Court will sign a judgment, pursuant to La. C.C.P. Art. 593 (D), upon the completion of the Phase III trial, and designate the said judgment as appealable pursuant to La. C.C.P. Art. 1915, as amended.

Subsequent trials will be set to determine and assess individual damages not common to the class in accordance with La. C.C.P. Art. 593.1 (C)(4)(1984).

The Court adopts Case Management Order No. 2 attached. Such order shall control the progress of all pre-trial proceedings. The provisions of Case Management Order No. 2 shall be followed strictly and are not subject to change by agreement of counsel without the permission of the Court.

All discovery disputes are hereby referred to the Special Master, as set forth in Case Management Order No. 2. The Special Master, who will conduct hearings and conferences, will make arrangements for the stenographic recording of all such hearings and conferences. The Special Master shall hear all discovery disputes expeditiously and shall file his report and recommendations as soon as is practicable after each hearing or conference.

The Court sets the following special hearing dates for all matters connected with this case. All hearings will be at 10:00 a.m.:

March 22, 2000	August 15, 2000
April 24, 2000	September 28, 2000
May 24, 2000	October 26, 2000
June 28, 2000	November 22, 2000
July 18, 2000	December 6, 2000

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The Court shall issue a formal Pre-Trial Order form as a guideline for the confection of a final joint Pre-Trial Order which will control the trial of this case. The Pre-Trial Order shall be filed in the record ten (10) days before the final Pre-Trial Conference.

The final Pre-Trial Conference is set for Thursday, December 13, 2000 at 10:00 a.m.

New Orleans, Louisiana, this 24-11 day of February, 2000.

Richard J. Ganucheau
JUDGE, Division "K"

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HAROLD BURNLEY
RELIANCE DOCUMENTS

Memo from E. Chambers to V. Willis Re: Cigarettes for Special Study (6/17/98)

Park 500 Process 30-Day Material Balance (7/15/96)

Letter from Helmut Gaisch to R.B. Seligman (9/12/83)

Report by I.L. Uydess & R.L. Dunn, "Mixed Culture Dentrification: Routine Storage and Start-up Procedures for Refrigereated, Denitrified Seed" (8/29/83) (1000417476-1000417492)

Report by I.L. Udyess, "Project 1904 - Annual Report" (4/22/83) (1003180085-1003180123)

Memo from K.H. Cho Re: Project Cancellation - N.O.D. Plant - Park 500 (4/27/83) (2060329239)

Memo from J.C. Gomes to H.G. Burnley, Jr. Re: Status Report - N.O.D. Project (3/19/82) (2022201031-2022201038)

U.S. Patent No. 3,420,241, Method of Preparing a Reconstituted Tobacco Sheet Employing a Pectin Adhesive (1/7/69)

U.S. Patent No. 3,353,541, Tobacco Sheet Material (11/21/67)

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PHILIP MORRIS U.S.A.

INTEROFFICE CORRESPONDENCE

Richmond, Virginia

To: Vivian Willis Date: June 17, 1998

From: Elizabeth Chambers

Subject: Cigarettes for Special Study

Data are given for the five cigarette samples submitted for a special study.

	Marlboro Lights KS	Marlboro Lights KS Configuration With 24% RLTC	Marlboro Lights KS Configuration With 24% RL150B
Smoke (FTC)			
Butt Length, mm	34	34	34
Tar, mg/cig	10.7	10.4	10.3
Nicotine, mg/cig	0.80	0.77	0.76
Puffs/cig	7.9	7.8	7.9
Tar/Puff, mg	1.3	1.3	1.3
CO, mg/cig	12.1	12.1	11.8
pH	5.30	5.30	5.30
NH_3, ug/cig	16.4	8.9	13.1
Cigarette			
Total RTD, mm of H_2O	118	122	121
Length, mm	84.0	84.1	83.9
Circumference, mm	24.8	24.8	24.8
Paper			
Permeability, CORESTA Units	25	26	27
Filter			
RTD, mm of H_2O	98	98	98
Length, mm	27.1	27.1	26.9
Tipping Paper Length, mm	31.0	31.0	31.0
Ventilation, %	19	20	21
Filler			
Total Alkaloids, % (DWB)	1.97	1.98	1.96
Total Red. Sugars, % (DWB)	8.1	8.0	8.0
Reconstituted Tob., %	19	24	25
Expanded Stems, %	2	3	2
Expanded Tobac %	11	11	10
BBO, %	1	None	1
Lamina, %	67	61	62
Wt of Tob., g (12.5% OV)	0.675	0.678	0.680
Rod Density, g/cc	0.243	0.243	0.243
Rod Length, mm	56.9	56.9	57.1
pH	5.79	5.66	5.75
NH_3, % (DWB)	0.36	0.18	0.30
Humectants			
PG in Filler, %	0.9	0.9	1.0
Glycerin in Filler, %	2.6	2.7	2.7

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	Marlboro Lights KS Configuration With 24% RL (no ingredients added)	Marlboro Lights KS Configuration With 24% RL (no ingredients added, no casing, no A/C)
Smoke (FTC)		
Butt Length, mm	34	34
Tar, mg/cigt.	10.0	9.4
Nicotine, mg/cigt.	0.78	0.82
Puffs/cigt.	7.4	7.4
Tar/Puff, mg	1.3	1.2
CO, mg/cigt.	11.0	10.2
pH	5.22	5.31
NH_3, µg/cigt.	7.9	6.7
Cigarette		
Total RTD, mm of H_2O	112	119
Length, mm	84.0	84.0
Circumference, mm	24.7	24.8
Paper		
Permeability, CORESTA Units	26	25
Filter		
RTD, mm of H_2O	97	98
Length, mm	27.1	27.2
Tipping Paper Length, mm	31.0	31.0
Ventilation, %	21	24
Filler		
Total Alkaloids, % (DWB)	1.94	2.20
Total Red. Sugars, % (DWB)	7.5	6.0
Reconstituted Tob., %	23	22
Expanded Stems, %	3	3
Expanded Tob., %	14	15
BBO, %	1	1
Lamina, %	60	60
Wt. of Tobacco (12.5% OV)	0.681	0.668
Rod Density, g/cc	0.245	0.240
Rod Length, mm	57.0	56.8
pH	5.57	5.55
NH_3, % (DWB)	0.16	0.18
Humectant/Plasticizer		
PG in Filler, %	0.6	ND
Glycerin in Filler, %	2.2	0.7

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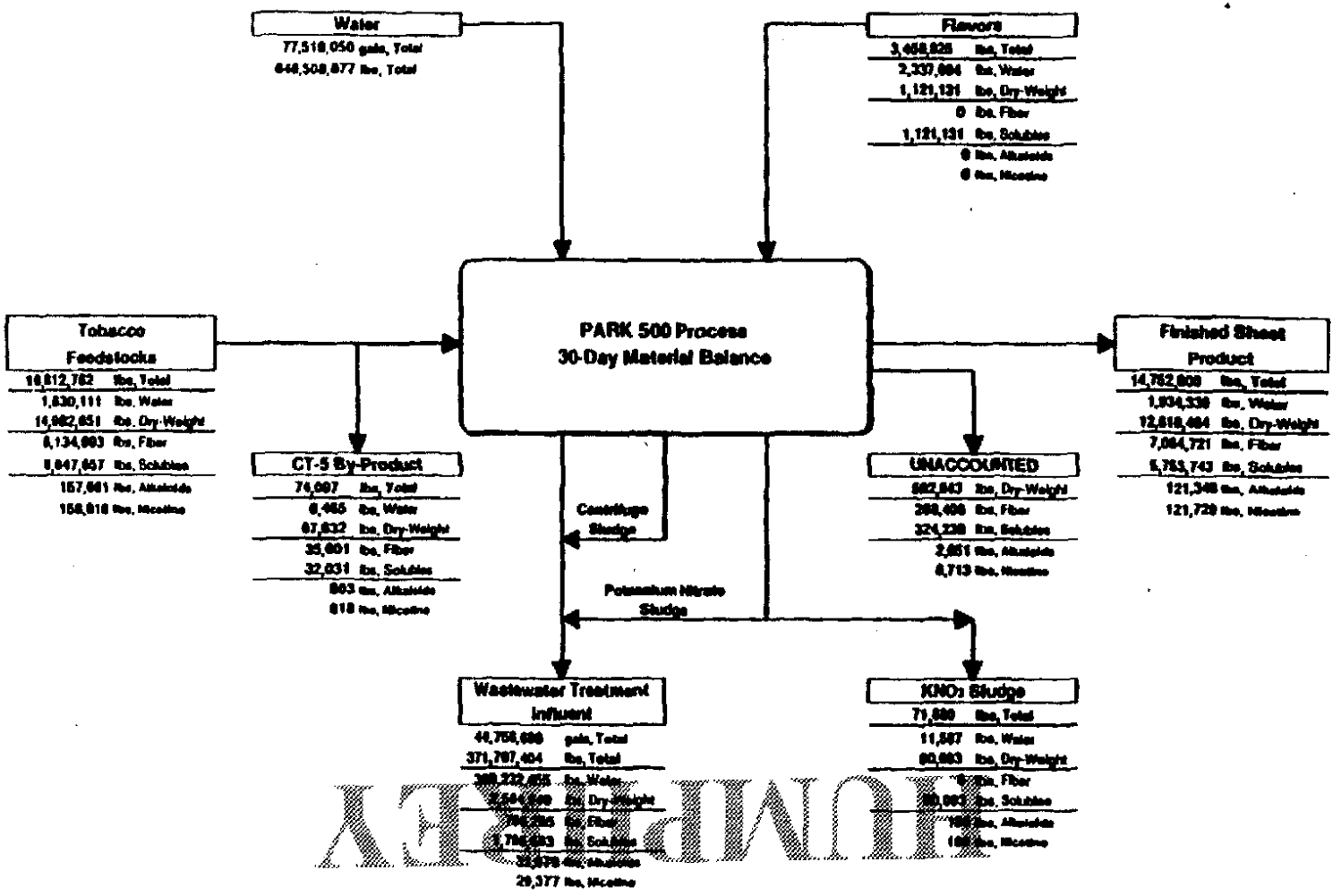

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Based of Park 500 Production from August 23 to September 21, 1995

(lbs, dry-weight)	FEEDSTOCKS	FLAVORS	PRODUCT	LOSSES	% LOSSES
TOTAL	14,982,651	1,121,131	12,818,464	3,285,317	20.4%
TOTAL w/o Flavors	14,982,651		11,810,014	3,172,638	21.2%
COMPOSITIONAL BREAKDOWN					
FIBER	8,134,993		7,064,721	1,070,272	13.2%
SOLUBLES	6,847,657	1,121,131	5,753,743	2,215,045	27.8%
FLAVORS		1,121,131	1,008,450	112,681	10.1%
W/O FLAVORS	6,847,657		4,745,293	2,102,364	30.7%
ALKALOIDS	157,661		121,348	36,313	23.0%
NICOTINE	158,816		121,729	37,088	23.4%
KNO_3	1,047,842		412,131	635,711	60.7%
"KNO_3 DISCARD"			635,711		
VALUES ADJUSTED FOR "KNO_3 DISCARD"					
W/O FLAVORS	6,847,657		5,381,004	1,466,653	21.4%
ALKALOIDS	157,661		123,255	34,406	21.8%
NICOTINE	158,816		123,636	35,180	22.2%

Value based on historic feedstock data for potassium nitrate.

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Park 500 Process - 30-Day Material Balance

Category (lbs, dry-weight)	INPUTS		OUTPUTS				DELTA
	FEEDSTOCKS	FLAVORS	PRODUCT	CT-5	KNO3 SLUDGE	WWT INFLUENT	INPUTS-OUTPUTS
TOTAL	14,982,651	1,121,131	12,818,464	67,632	60,093	2,564,949	592,643
SOLUBLES	6,847,657	1,121,131	5,753,743	32,031	60,093	1,798,683	324,238
FIBER	8,134,993	0	7,064,721	35,601	0	766,265	268,406
ALKALOIDS	157,681	0	121,348	803	180	32,679	2,651
NICOTINE	158,816	0	121,729	818	180	29,377	6,713
WATER	*1,836,111*	*2,337,694*	*1,934,336*	*6,485*	*11,587*	*389,232,455*	*-387,017,037*
						Process Water Input	*646,508,877*
						Water Delta	*279,491,840*

	% YIELD				ACCOUNTABILITY
	PROD/FEED	FF-PROD*/FEED	WASTE**/FEED	FLAV OUT*/FLAV IN	OUTPUTS/INPUTS
TOTAL	85.6%	78.8%	17.5%	89.9%	96.3%
SOLUBLES	84.0%	69.3%	27.1%	89.9%	95.9%
FIBER	86.8%	86.8%	9.4%		96.7%
ALKALOIDS	77.0%	77.0%	20.8%		98.3%
NICOTINE	76.6%	76.6%	18.6%		95.8%

* Flavor Free-PRODuct adjusted for flavor content; composite flavor level = 7.87%

** WASTE includes KNO3 Sludge and Wastewater Outputs

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R. B. SELIGMAN

Dr. R.B. Seligman
Vice President
Tobacco Technology Group
PHILIP MORRIS USA
P.O. Box 26583
RICHMOND, Va. 23261 / USA

HGA/jig

12th September, 1983

C83-09190

Dear Bob,

Thank you for your letter, in particular the first paragraph.

I had a good look at the NOD paper which contains a lot more information than had been compiled before. In the introduction the authors say for example "A complete understanding of the relationship between the relative efficiency of denitrification and the variability of our SEL's lies somewhere in the future". I think this sums it up. There are many conditions outlined which must be observed, but they are not terribly well defined and seem, to me at least, not easy to be controlled experimentally.

The most important feature of any industrial process is its controllability. Parameters which spring to mind are temperature, p_H, specific nutrients, etc. I know that the process runs, and I know that biochemically the interactions between two cultures are rather complicated. Where I see a problem, but I might be wrong, is how to keep the process on the rails. For example, what do you do if

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the start-up does not happen or what do you do if suddenly you accumulate the wrong metabolites ? However, the people who work with the system must have a feeling of how well they can control it. They are the only ones who can really judge.

I am asking myself why the process is conceived to be running in the fed-batch mode. The system is obviously fairly sensitive to fluctuations in the composition of the medium. In order to minimise this adverse influence, it was decided to leave a 30 percent inoculum level for re-starting. P_H control, the exclusion of oxygen, and so on have to be done on the remaining two thirds of the (fresh) medium in order to guarantee continuity in the wanted metabolic fashion. If, on the other hand, one would run in the continuous mode, wouldn't the corrections be only minimal and wouldn't the fluctuations be smoothed out ? At the same time P_H control and essential nutrient adjustment (additives) would give one a certain leaverage to steer the process down the right lane. Also in terms of P_{Oxygen} there should be an advantage, because once having reached the low level necessary for anaerobic metabolisation, the partial pressure would never rise again and could certainly not fluctuate into dangerous zones.

I hope to see you soon.

Kind regards and best wishes,

Helmut Gaisch

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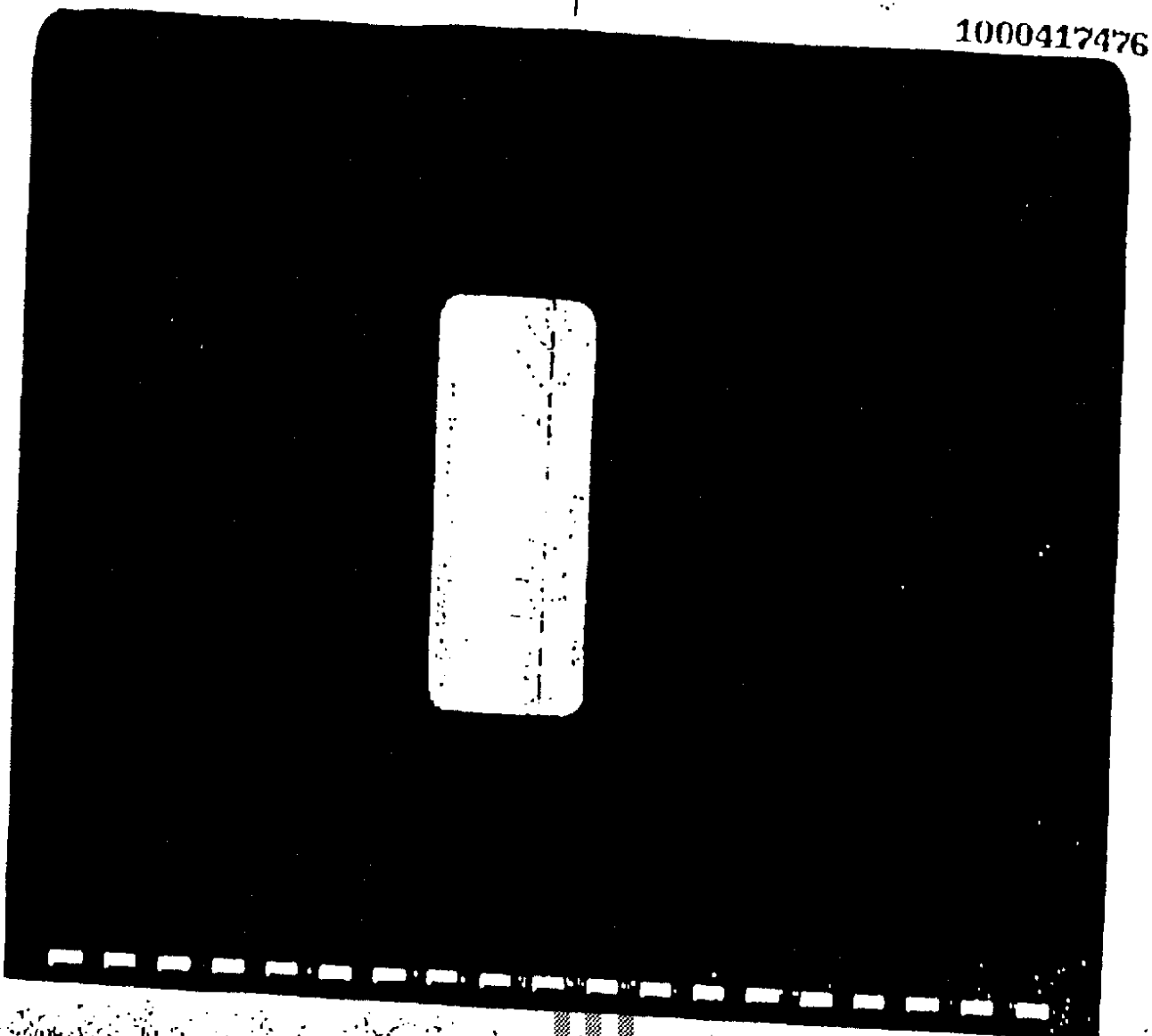

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CHARGE NO. & TITLE:

TYPE REPORT: ○ ANNUAL ○ SEMI-ANNUAL ○ COMPLETION ⊗ SPECIAL

DATE: August 29, 1983 PERIOD COVERED:

REPORT TITLE: Mixed Culture Denitrification: Routine Storage and Start-up Procedures for Refrigerated, Denitrified Seed.

BY J.L. Uydess R.L. Dunn

APPROVED BY J.K. Whidby

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KEYWORDS: NDD, Dissimilatory Denitrification, Nitrate, Nitrite, CEL, SEL, AU, BL, Microbiology, Biotechnology, Tobacco Processing.

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SUMMARY

Straightforward and reliable storage and start-up procedures are described for the NOD mixed culture seed. The denitrifying product from semi-continuous fermentors is collected with a small residual level of Nitrite still present in the effluent (1-200 ppm NO_2^- ; 0 ppm NO_3^-). The collected material is then immediately refrigerated at 0-4°C. The residual Nitrite present at the time of collection is completely reduced to nitrogenous gas by the time the stored seed cools to 15-20°C, the temperature at which active denitrification ceases[1].

The principle advantage of this storage modality is that the storage form itself is the unmanipulated product of this process and requires no extraordinary preparative manipulations. However, unlike a "dry", low maintenance storage technique such as lyophilization, which renders the dehydrated product more-or-less stable, the activity of the hydrated, refrigerated seed changes with time. This change is manifested primarily as a progressive increase (with storage time) in the time taken to completely denitrify fresh SEL once that SEL has been inoculated with the refrigerated seed. Typically, seed stored at 0-4°C for up to two weeks and then used as a 30% v/v inoculum will denitrify that volume of SEL (NO_3^- = 2000 ppm) within 8 to 16 hours under ambient conditions[2]. Seed stored for 6 months, or more, may take anywhere from 30 to 60 hours to denitrify, depending upon the initial viable cell count and quality of the feed SEL. Sparging fermentors with N_2 prior to and after inoculation reduces the "time to start" by 10 to 50%, again depending upon storage age, viable count, and SEL quality. Generally, however, the older the seed, the greater the percent reduction in start-up time with sparging. Thus, the comparatively retarded activity of older seed does not appear to be due solely to a loss in viability with time, but also (and possibly primarily) to a reduced capacity to induce the required, O_2 - sensitive denitrification enzymes in the presence of an increasingly oxygen-saturated environment. Once the initial startup has been completed, however, process operation (with regard to D-rates, cycle times, etc.) is the same for both "young" and "old" seed-initiated reactors.

While refrigeration of "active" liquid product has been shown to be an effective and easily recoverable storage modality for routine use in the short term, a more stable "dry" storage methodology such as lyophilization would be better suited for securing the integrity of the NOD process in the long term.

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[1] The mixed culture continues to respire Nitrate and Nitrite at a very slow rate even at 0-4°C ("maintenance respiration" - see following report).

[2] No measures are taken to drive the system anerobic (by sparging, etc.).

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Introduction:

The complete dissimilatory denitrification of SEL under anaerobic conditions has been consistently demonstrated over recent months in both laboratory (1) and Pilot Plant Fermentors (2) using the mixed culture of microorganisms orginally isolated from similarly denitrified SEL one year ago (3). Thanks to the collaboration of Dr. Walter P. Hempfling, we now know the principle participants to be an ATCC-1-like, Gram (+) Bacillus (species unknown), and a Gram negative rod, Alcaligenes faecalis var. denitrificans (4). Both organisms are native to soil and are tobacco identical. Figure 1 diagramatically depicts the proposed respiratory and metabolic relationships between these two bacteria. As can be seen, the Bacillus appear to be the primary Nitrate utilizers (and Nitrite producers) while the Alcaligenes are the predominant (sole?) Nitrite reducers.[1] Each of these organisms share several general environmental and physiological requirements which permit them to function and to participate coordinately in this process (as they undoubtedly do in the soil, or on the tobacco leaves in their native habitat). However, each of these organisms also has specific capabilities, requirements and/or sensitivities unique unto itself. An appropriate minimal set of acceptable circumstances must exist for each organism to function successfully with respect to mediating the complete and consistent dissimilatory denitrification of SEL at a "reasonable" rate. When one (or more) of these environmental or nutritive conditions is not right, their adverse impact on each of these organisms, and thus on the normal phenomenology of the NOD process can be observed as either a deminished capacity to convert Nitrate to Nitrite, Nitrite to gas, or both.

Over the past six months we have made significant advances in our understanding of the major operational and process parameters of NOD (5,6). However, one of our greatest problems has been in gaining a better understanding of the critical parameters regarding seed storage and a consistent process start-up. We now know that the metabolic "acceptability" of the substrate is one of the most crucial factors in this process (as it would be in any other). Unlike a pure culture (single isolate) system, in which a single set of nutritive and environmental parameters satisfies the entire homogeneous microbial population, our mixed culture system, composed of at least two significantly dissimilar microorganisms, necessitates that multiple sets of minimally-acceptable environmental and nutritive parameters be provided. Although considerable light has recently been shed on this topic as a result of the recent experimental observations of the NOD laboratory and Dr. Hempfling, et. al., (4) a complete understanding of the relationship between the relative efficiency of denitrification and the variability of our SEL's lies somewhere in the future.

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[1] The Alcaligenes are also capable of reducing NO_3^- to NO_2^- as well (personal communication from Walter P. Hempfling).

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FIGURE 1

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While an increased level of understanding of these relationships has definately advanced our ability to control and implement our semi-continuous denitrification process, we have not had, until recently, a good handle on the critical parameters necessary for short and long term storage of the mixed culture, nor have we been able to demonstrate repeatably and reliably an efficatious and rapid start-up procedure.

In October, 1982 it became apparent that refrigerated (denitrified) product stored for 15 months at 0-4°C could be used successfully as inoculum to re-start the NOD process in fresh set (3). However, the time necessary to "start" was slow (>48 hours) and multiple trials were inconsistent. But this material did start, and it was from this point which we began our study of the optimization of the refrigeration of bulk, liquid product as a short-to-moderate term storage modality.

Remember, one of our principle project goals was to investigate alternative seed storage methodologies and to define effective start-up procedures for the NOD seed (our previous inability to demonstrate controlled process start-ups was one of the most significant shortcomings we had to overcome). As a result, a start-up procedure was needed which would be:

1. Straightforward - involving a minimum of special manipulation or methodology.
2. Dependable - having a high probability of success.
3. Reproducable - that each start-up had similar results.
4. Predictable - that the time-course and phenomenology of each start-up was predictable based upon a large data-base of previous start-up results under widely varying conditions of substrate and seed, and,
5. Rapid - that the start-up procedure of choice allowed for as rapid a start-up as possible within the confines of the microbiological and chemical systems employed.

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Results and Observations:

We began by refrigerating multiple, 2-liter aliquotes of freshly denitrified product for a period of several days to several months. The refrigerated product was then removed from the coldroom at various intervals (representing different durations of storage) and added as a 10% v/v inoculum to sterile SEL feeds adjusted to pH 5.8 to 6.0 and held at 46-47°C prior to inoculation (5). The time to start[1] was generally poor, (even for "fresh" inoculum) and in the range of 36 to 48 hours (or more). However, our continued experience running the mixed culture in both semi-continuous and batch fermentors soon elucidated to us the mixed culture's rather stringent requirements for a minimal sustained level of excess respiratory substrate (Nitrite) during process operation, as well as during collection of product for future seed (1,6,7).

Experiments conducted in the coldroom on freshly-collected (46°C) product containing excess levels of Nitrate and/or Nitrite demonstrated that significant levels of these substrates were respired during cool-down of the culture. Typically, 2-300 ppm of Nitrate/Nitrite could be reduced to gas during the first 1/2 to 2 hours of cool-down, or, until the temperature of the mixed culture was recuced to approximately 25°C.[2] For this reason, the NOD seed was always collected with a residual NO_2^- level of at least 100-200 ppm's (when possible) to avoid loss of this denitrification capacity (re-repression of the dissimilatory denitrification enzymes).

However, simply satisfying the residual respiratory substrate requirement of these organisms alone was not enough to guarantee fast, repeatable and dependable start-ups from refrigerated seed. There was something else.

---

1 The time from inoculation to the complete denitrification of the initial (starting) "batch" volume of feed SEL in the fermentor.

2 Remember, these organisms are native to the soils of many of these temperate tobacco-growing regions where they grow under widely varying climatic conditions.

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Several rather blatent operational mistakes then became apparent to us. Unwittingly, we had been subjecting our stored inoculum to two rather severe environmental shocks during our start-up procedure. Firstly, we were instantaneously introducing cold (0-4°C), dormant seed into a comparatively "hot" environment (46-47°C) and then asking it to "perform". This sudden thermal transition alone was certainly enough to perturb the mixed culture.[1] Above and beyond this, we were also rapidly introducing a small volume of stored seed at a pH of 7.6 to 8.2[2] into a larger, bulk environment of feed SEL at a pH of 5.8 to 6.0, (another significant environmental shock[3]).

Realizing this, we modified our start-up procedures to avoid (as best as possible) both these abrupt changes in temperature and pH (8).

Current Methodology

Denitrified seed, stored for some variable period (days to months) at 0-4°C, is now allowed to warm naturally at room temperature to between 15° to 20°C and is then introduced into a sterilized "cold" volume of SEL feed held at 15° to 20°C. The feed SEL has also been adjusted to pH 7.6 to 8.0 to match the "finishing pH" of the stored seed (see discussion of the relationship between denitrification and pH, reference 6). Furthermore, the percent inoculum used has been increased (from 10%) to 30% v/v. Ten and twenty-percent inoculum start-ups (in air)[4] were observed to be quite variable with regard to the length of time necessary to start. It wasn't until 30% v/v inocula were employed that highly consistent and reliable start-ups were obtained routinely.

---

[1] There is a considerable body of literature on the effects of abrupt thermal transitions on the microbial cell cycle. The results range anywhere from mild delays in the timing of cellular events, to cell lysis.

[2] Remember, in the NOD process pH rises as denitrification goes to completion.

[3] Abrupt and significant changes in pH can have a manifold adverse impact on many macromolecular structures, especially on biological membranes, which ultimately participate in virtually all cell processes.

[4] Under ambient conditions, without sparging.

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The observed variability in start-up time using lower volume inocula under non-anaerobic conditions is indicative of a phenomenon in which some minimum population of "competent" cells is required to successfully initiate and complete prerequisite tasks obligatory to this anaerobic dissimilatory denitrification process (e.g. O_2 removal). Oxygen is known to differentially inhibit the synthesis of the enzymes of this pathway, especially of Nitrite Denydrogenase, which mediates the reduction of NO_2^- to gas (9,10,11).

The adverse impact of oxygen on the progression of denitrification in "batch" systems using small numbers of cells has recently been demonstrated in this laboratory by J.P. Kusel (12,13). These data are consistent with our previously mentioned fermentor data and with the recent mixed culture model of Hempfling, which depicts the Gram Positive population of Bacilli as being the oxygen "scrubbers". It is envisioned that under non-anaerobic conditions, the Bacilli remove (respire) the residual dissolved oxygen thereby making it possible for the Gram Negative Alcaligenes to reduce NO_2^- to NO. Until this function is completed, Nitrate will be converted to Nitrite, but Nitrite will not be removed to gas. Purging the system with H_2, Ar_2 or He_2 drives the environment more anaerobic and alleviates some of the constraints placed on cell number (of the Bacillus sp.) greatly.

Armed with a greater understanding of the relationship between the mixed culture's capacity to denitrify and the state of their transition into and out of that environment, we set out to examine the relationship between start-up time and the "age" of the stored inoculum (storage age at 0-4°C). Approximately 80 "cold" start-up experiments were conducted using 30% v/v inocula under the experimental conditions outlined previously. The results from these experiments are given in Graph 1. The squares (□) indicate data from experiments in which there were known (documented) operational problems or mishaps resulting in artifically-long start-up times (including power failures and temperature controller failures).

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The circles (Ø) represent the JCI inocula, cold start-up data from the Pilot Plant (seed fermentors T-3 and T-4) during the recent NOD test runs of June-July, 1983. As you can see, the fit between "storage age" and the "time to start" is really quite good, especially in light of: 1) the chemical variability of our SELs, 2) the "biological" nature of the system, and 3) the variability in the initial cell number of the seed used (Product collected from actively denitrifying fermentors may contain anywhere from about 1 to 5 x 10^9 cells/ml depending upon the quality and original Nitrate level in the feed SEL). Graph 2 gives the "time-to-start VS seed age" data for equivalent aliquotes of inocula of the same storage age under "as-is" (+——+) and N_2 - sparged (+----+) conditions. In each case, the N_2 - sparged cultures started-up faster, again demonstrating the known requirement for anaerobiosis.

The form of the data for "as-is" (non-anaerobic) start-ups in Graphs 1 and 2 originally led us to believe that start-ups from "older" seed were primarily cell-number limited -- that there was a progressive loss in viability with increasing age of storage at 4°C. (That the longer start-up times experienced with "older" seed were a reflection of the increased time required to replace successively larger portions of the microbial population lost during long intervals of storage). However, the normalization of the data for the Nitrogen-sparged start-ups in Graph 2 argues more for re-repression (loss of capacity) of the Nitrite - removing enzymatic machinery than for loss of viability (although there may be a small loss in viability also - but not to the level that we had previously envisioned).

It is interesting to note that the data for all but one of the Pilot Plant start-ups cluster in the low range of start-up times for the ages of seeds used (e.g., the same seed started up faster in the Pilot Plant fermentors than in laboratory fermentors). The only significant difference in conditions between the laboratory and Pilot Plant is the fact that the Pilot Plant SEL is sterilized within the fermentors with clean steam. The steam, which is sparged through the SEL, sterilizes it, but also drives off dissolved gases, such as oxygen, thereby making the Pilot Plant sterilized SEL comparatively more anaerobic. This effect is more prominently observed in "older" seed which may have a greater saturation of dissolved O_2 and a somewhat deminished capacity to deal with it. Once a de novo start-up has been successfully completed and feeding of the denitrified "heel" is begun, normal semi-continuous process operation can be initiated (perpetuated) and the same nominal dilution rates obtained irrespective of the original storage age or start-up time of the seed.

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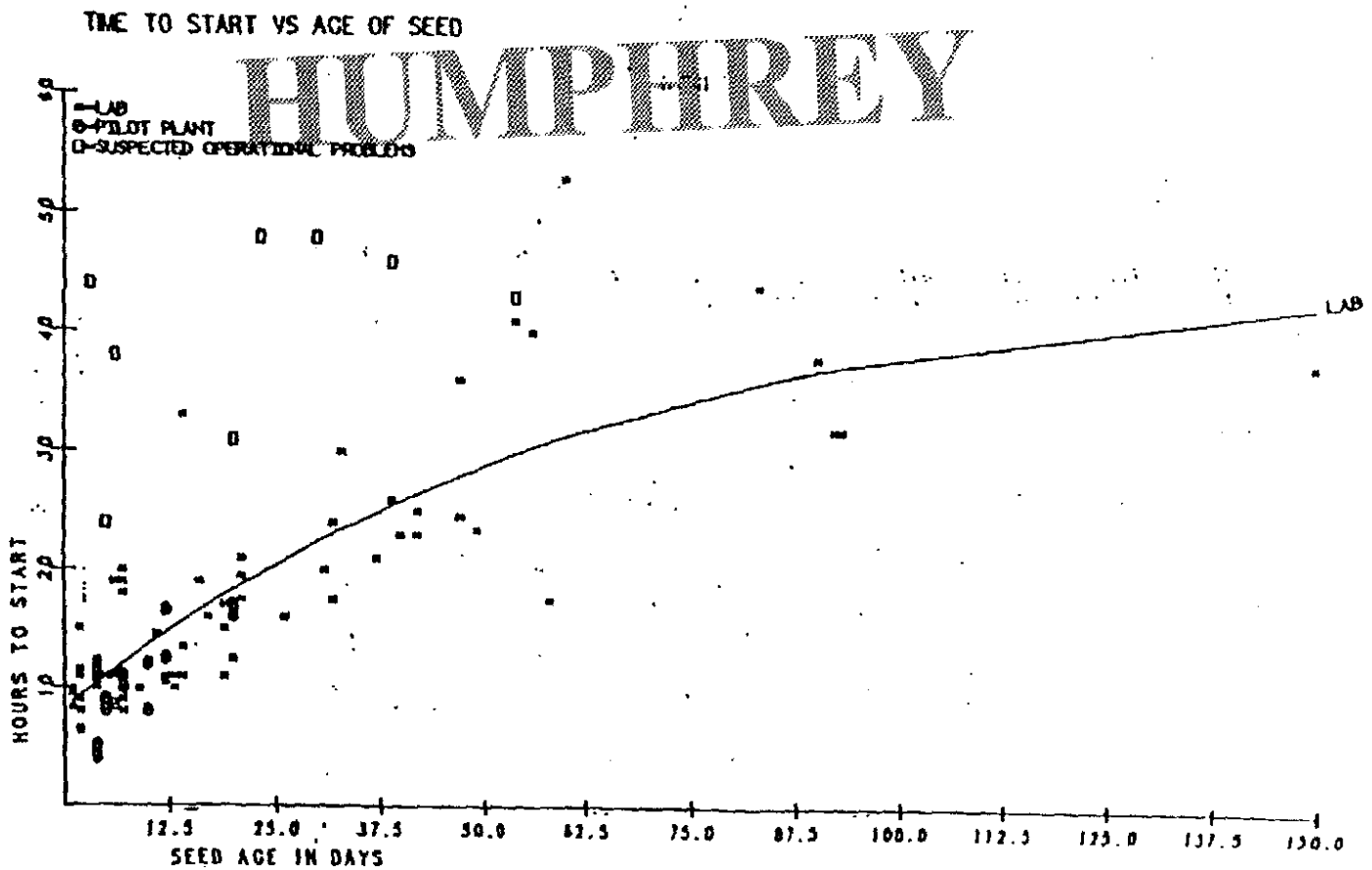

GRAPH 1

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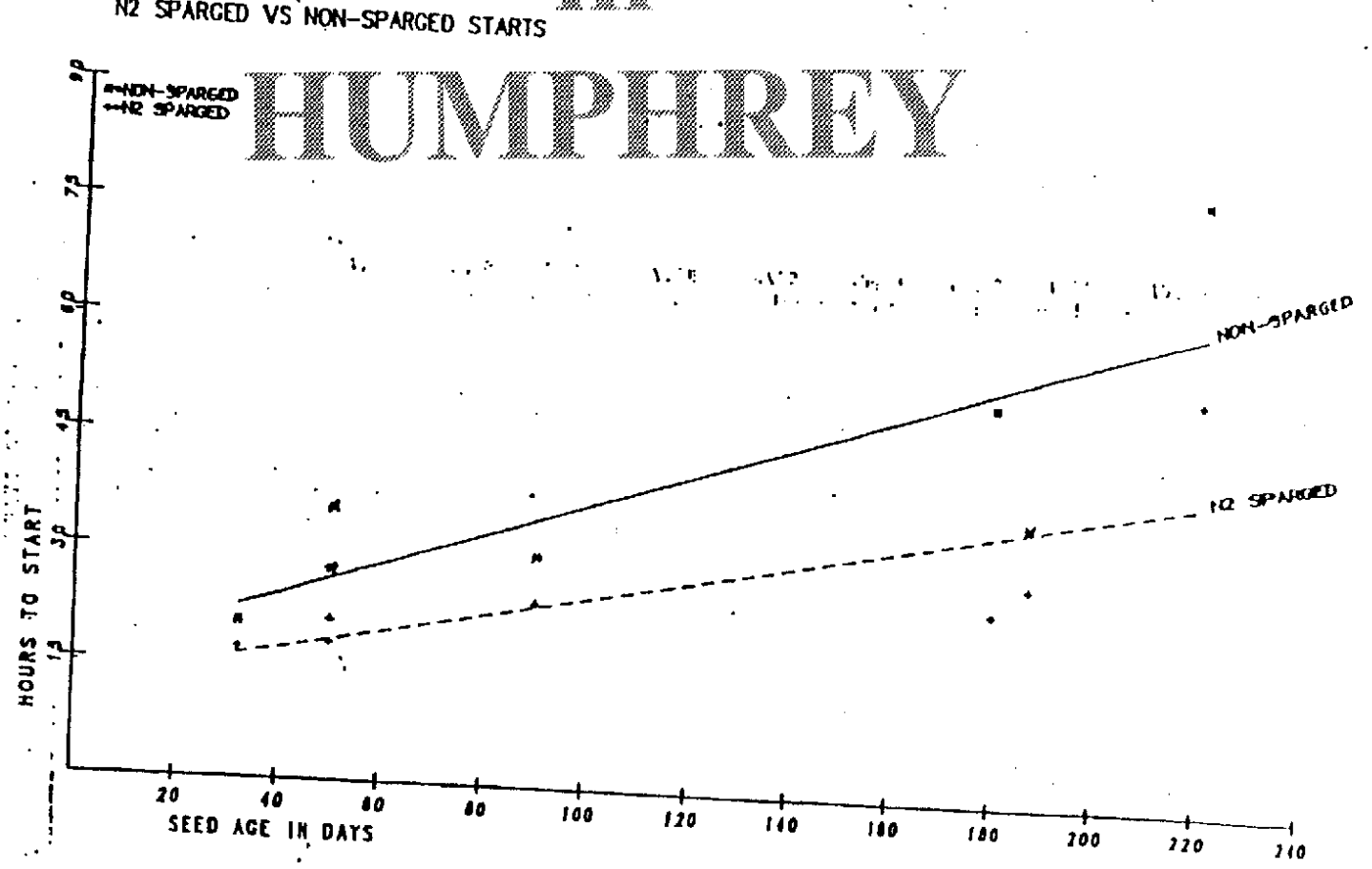

GRAPH 2

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Conclusions:

Now that the major environmental and metabolic parameters of the mixed culture are better understood, we have been able to demonstrate the utility of using the refrigerated, denitrified product from one denitrification run as seed for starting up subsequent denitrification runs. The time required to completely denitrify the original "batch" volume of feed SEL in such de novo start-ups has been shown to be related to the duration of storage at 0-4°C: the longer the duration of storage, the longer the interval to totally denitrify the initial batch volume of SEL.

Generally, under ambient start-up conditions, seed stored for 1 to 2 weeks will take 10 to 18 hours to start, while seed stored for 4-6 months will take 40 to 60 hours to start. Sparging the de novo start-up environment with N_2 results in a 10% to 50% reduction in start-up time, depending upon the original storage age of the seed. (For seed stored up to 6 months, this means a total start-up time of not usually more than 24-36 hours).

Although this storage modality may not prove to be the most efficient vehicle for long-term maintenance of the MCD seed, it does have several important advantages over other procedures.

1. The storage form is the natural (hydrated) product of this process and requires no special or extraordinary processing or manipulation to prepare it either for storage or for use a inoculum.

2. The storage form can be used directly as inoculum to (re)establish active denitrification under non-stringent (ambient) conditions in a rapid, dependable and straightforward manner.

3. The performance (denitrifying potential) of the stored product is known and predictable from: a) the storage age of that refrigerated culture, and b) the past performance of that culture in the fermentor(s) of origin (there is no reason to expect significant deviation from this past performance[1] since this storage modality is not subjected to any perturbatious manipulations).

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[1] In an equivalent "quality" SEL, that is.

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On the negative side, however, is 1) the long-term instability of this hydrated refrigerated material, 2) the requirement to regenerate fresh seed at frequent (monthly) intervals[1], and 3) its' suseptability to spoilage as a result of an interruption in refrigeration. Storage at 0-4°C does not preclude continued chemical activity at both the microbial and environmental (substrate) levels. Long-term Nitrate utilization studies in the coldroom have already demonstrated that the mixed culture will continue to slowly respire Nitrate and Nitrite at 0-[illegible] (presumably, maintenance respiration). Chemical modifications to the SEL also undoubtedly occur (remember, the pH of the refrigerated material is often in the range of pH 7.8 - 8.0). For this reason, and to otherwise "protect" the integrity of this process in the future (both in the short and in the long-term), a more stable and environmentally independent storage modality is required. In this context, lyophilization would appear to be the most efficatious and logical choice.

In lieu of this, J.P. Kusel has recently demonstrated that small volumes of mixed culture seed that have been successfully lyophilized as 1:10 dilutions of the original product in a defined (non-SEL) medium, can subsequently be used to start laboratory-scale fermentors after an initial re-hydration/re-activation step [14]. A joint program with an outside consultant/contractor will soon be undertaken to optimize this long-term storage modality and to produce certified, lyophilized seed for future NOD development.

[1] If rapid startups (8-12 hours) are desired.

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This work could not have been conducted without the assistance of the PM Temporary Employees who worked long hours to run and maintain these experiments: P. Finn, P. Boone, A. Conyers and M. Varnier.

Special thanks to H. Hartung for his instruction and continued guidance on the use of his EZ DATA and EZ PLOT programs, and to Pat Finn and Andrei Conyers for implementing these programs.

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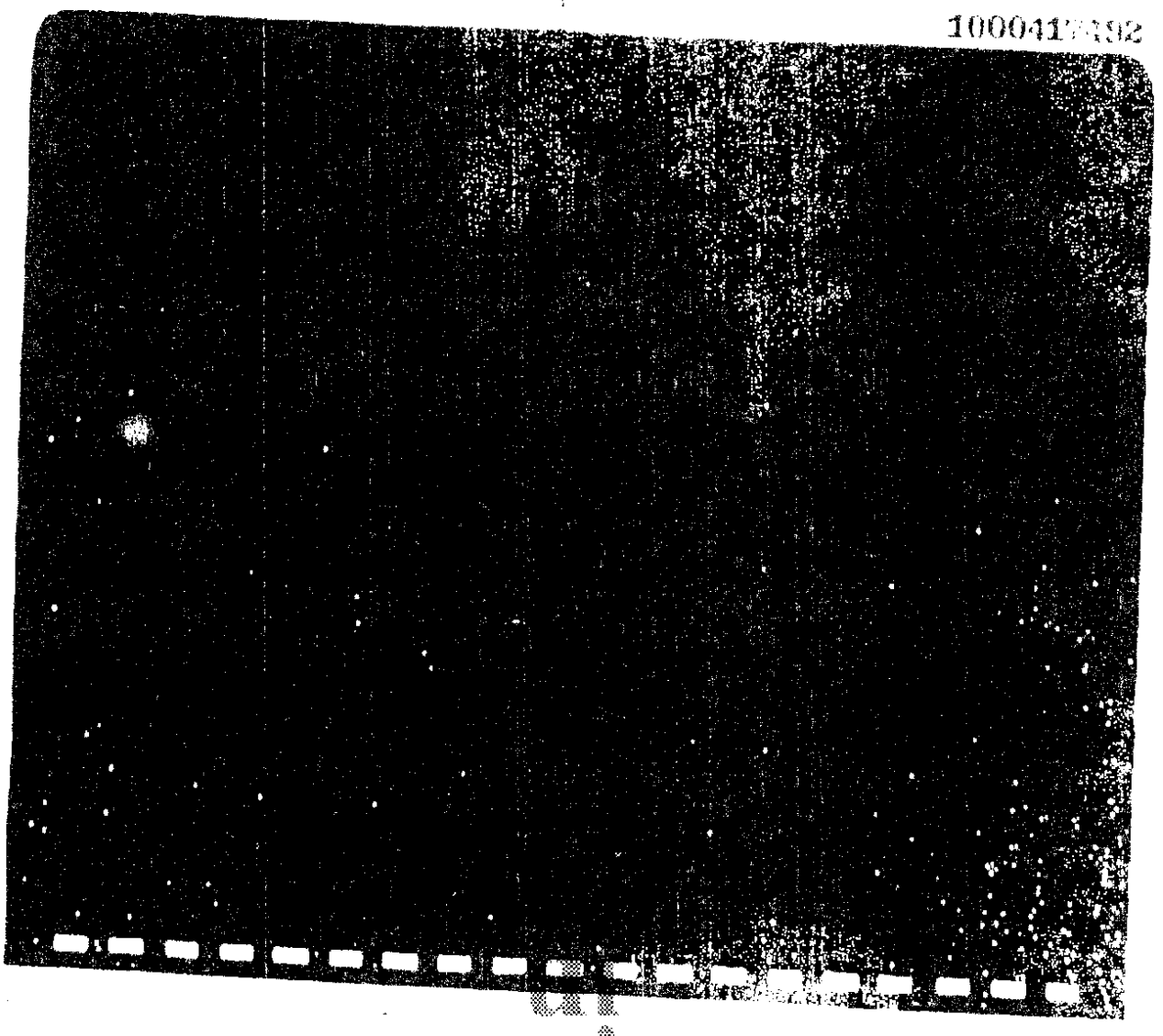

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ABSTRACT

The progress made to date to elucidate the phenomenological basis of, and operating and monitoring parameters for the Naturally Occurring Denitrification process (NOD) is discussed.

Organisms indigenous to tobacco have been shown to be capable of mediating the complete disimilatory denitrification of our strongly extracted tobacco liquors (SEL) at industrially-acceptable dilution rates of >10%. Although considerable effort was expended examining the denitrification potential of a single isolate pure culture (ATCC-1), the mixed population of bacteria originally isolated from our spontaneously-denitrifying SELs has been shown to be more reliable and less fastidious with respect to its use in an industrially-scaled process.

Operational, process, and monitoring parameters are discussed in light of laboratory-scaled fermentation experiments. The optimization of start-up, temporary hold and shut-down procedures is also discussed.

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INTRODUCTION:

An historical overview of the Naturally Occurring Denitrification process (NOD) has previously been given.[1] Although the original observations upon which this process was based were shown to be the result of the activity of a mixed population of bacteria, subsequent process development aspirations re-directed the laboratory effort toward identifying and utilizing a single, pure culture isolate upon which an industrially-acceptable process could be designed. A thermotolerant Bacillus (sp. unknown) isolated from SEL by the American Type Culture Collection and alleged to be a potent solitary denitrifier[2] became the focus of the NOD laboratory investigations. Laboratory and Pilot Plant operating procedures were modified to incorporate "aseptic operation" to accommodate working with the pure culture. However, a number of important questions persisted with regard to: 1) the true purity of the ATCC isolate,[3] 2) the isolate's inherent capacity to function as a potent and solitary denitrifier,[4] and 3) the possible perturbations suffered by the isolate as a result of the methodology employed during isolation and lyophilization at ATCC. Laboratory and Pilot Plant experiments failed to verify conclusively, that the pure culture that was supplied to PM by ATCC was indeed capable of reproducible start-ups and complete denitrification. As a result, our attention turned once more toward the mixed culture from which this isolate had originally been obtained. Subsequent experiments with both the mixed culture and freshly-isolated "pure" cultures of ATCC-1-like strains of Gram + and Gram "variable" bacilli repeatedly demonstrated that 1) the mixed culture was apparently a more reliable and more potent mediator of

1. Banyasz, J., Proj. 1904 Annual Report, May 21, 1982, Accession #82-159.
2. Ibid.
3. Op. cit. p. 3, and recent observations.
4. Uydess, I. L., "Denitrification," memo to J. Banyasz, October 7, 1982.

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complete dissimilatory denitrification than ATCC-1, and 2) that while ATCC-1-like organisms may be capable of complete denitrification under certain conditions, we didn't understand enough about the (unique?) requirements of this elusive capacity to utilize it within the confines of our physical planning to develop, engineer and implement an industrially acceptable and commercially feasible NOD facility for PM USA. In lieu of this, we preceeded with our investigations using the mixed culture. However, several crucial questions still had to be answered:

1. Could the mixed culture be proven to be a reliable and potent denitrifier?,
2. Could straightforward, dependable (predictable) start-ups be demonstrated from "stored" seed?,
3. Would the mixed population remain stable over long periods of growth and denitrification with regard to:
   a. it's microbial composition (population consistency),
   b. it's ability to sustain active denitrification over long periods of time, and,
   c. the quality (variability) of its product?
4. Could an industrially acceptable process be demonstrated (e.g. >10% dilution rates) using the mixed culture? and,
5. Could the mixed culture cope with the variability in quality (composition) of our SELs?

The following report summarizes the progress made in answering these questions over this past year. Portions of this document are abbreviated due to the detailed coverage of some of these topics in recently issued special reports and memos which are referenced or appended herein.

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RESULTS AND OBSERVATIONS:

A. THE BACTERIA:

The Naturally Occurring Denitrification process (NOD) as originally observed and documented by B. Semp[5] at our Park-500 RL facility, was shown to be mediated by a mixed culture of bacteria. Our own light and electron microscopy observations confirmed this, and further elucidated that the mixed population was apparently composed of both Gram (+) and Gram (-) rods of diverse morphologic forms, as well as some Gram (+) cocci and spores.[6] At about that same time, the American Type Culture Collection facility (ATCC) isolated and partially identified four Gram (+) and/or Gram variable bacilli (species unknown), one of which (ATCC-1973; our ATCC-1) appeared to be the predominant denitrifier.[7] However, subsequent investigations within PM R&D were not able to consistently show that this lyophilized pure culture as supplied to us by ATCC was a reliable mediator of the complete denitrification (to nitrogenous gases) of our SELs.[8] An immunofluorescence assay employing specific antibody made to ATCC-1 developed by PM R&D personnel[9,10] did, however, confirm that this bacterial (antigenic) type was a major component within the mixed population. However, such tests as these, although potentially powerful tools, only verify the genetically-specified antigenic "fingerprint" of an organism's surface, and not the physiologic capacity of the organism itself.

5. B. A. Semp, "Exploitation of Naturally Occurring Fermentation," memo to C. H. O'Donohue, Nov. 19, 1980.

6. I. L. Uydess, EM observations, Lab Notebook 7645, 1981.

7. Malik, V. S., personal communication(s), 1982.

8. J. Banyasz, Proj. 1904 Annual Report, Acc. No. 82-159, 1982.

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As our investigations shifted to an examination of mixed-culture mediated denitrification, we recognized the need to verify, as best as we could, the composition and stability of this heterogeneous mixture of bacteria during prolonged periods of growth and denitrification. However, our in-house tools and expertise were not particularly extensive in these areas. As a result, we initially had to rely upon rather limited characterization techniques, including a) light microscopy, b) the Gram stain, c) colony morphology on agar plates, and d) a bank of biochemical test media, all in conjunction with our daily observations and data on the "quality" of denitrification in whatever experimental situation was being examined at the time. However, within the confines of the relatively simple tools at hand, we were able to verify that the overall quality of the mixed culture (with respect to the apparent number of phenotypically-different participants therein) had not changed significantly oven the past four months of more-or-less continuous growth and denitrification in laboratory-scale fermentors. Biochemical, cultural and morphological observations coupled to more recent immunofluorescence assays continue to show the presence of Gram (+) to Gram variable ATCC-1-like species which comprise a major portion of the total bacterial population. Additionally, the Gram stain and various other morphologic, cultural and biochemical tests also demonstrate the continued presence of both Gram negative and Gram-variable rods, and apparently, Gram (+) variable cocci. Each of these morphologic types have recently been isolated and partially characterized by Dr. Walter P. Hempfling,[11] a collaborator and consultant to the current NOD Bioengineering program. Additional information on the taxonomic identifications and physiologies of these organisms will be made available once the preliminary findings have been substantiated.

11. Department of Biology, Univ. Rochester, Rochester, NY.

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3. THE PHENOMENOLOGY OF THE PROCESS:

1. Calculation of Equivalent Dilution Rate in a Semi-Continuous System

Before presenting a description of the process itself, it would be helpful to first set forth and explain how we calculate Dilution Rate (D-rate) in the laboratory. The term "Dilution Rate," as it is used in the bulk of the microbiology literature, primarily addresses itself to a description of the throughput (or fractional volume changes per hour) of a continuous culture system having a fixed (non-varying) total working volume (such as a Turbidostat or Chemostat). The formula for calculating the "D" rate of such a system has conventionally been given as: $D = f/v$ where "f" is the flow rate of substrate per hour (in and out of the system), and "v" is the fixed working volume of the system.[12] This also implies that there is a fixed population of microorganisms - the exact number being defined by the specific growth rate under those conditions of substrate and environment. In this program, however, we knew that we would be dealing with a semi-continuous "fed batch" system in which a given volume of fresh substrate would be added over some defined period of time to "X-volumes" of a previously processed substrate ("heel") containing the active culture of bacteria. This, then, specifies two major departures from classical continuous culture methodology.

First, we would now be dealing with a continuously changing (increasing) working volume, and second, a necessary and more-or-less concomitant increase in the total "working" population of bacteria. Thus, our "black box" isn't constant, but dynamic. (We would, in effect, be adding X-volumes of fresh substrate per unit time to an ever-increasing working volume; dv/dt from T_o to T_{final}). As a result, we decided to modify the classic dilution rate formulation to better suit our operational and functional needs - after all, our primary

12. Herbert, D., et al., J. Gen. Micro, (14) 601-622, 1956.

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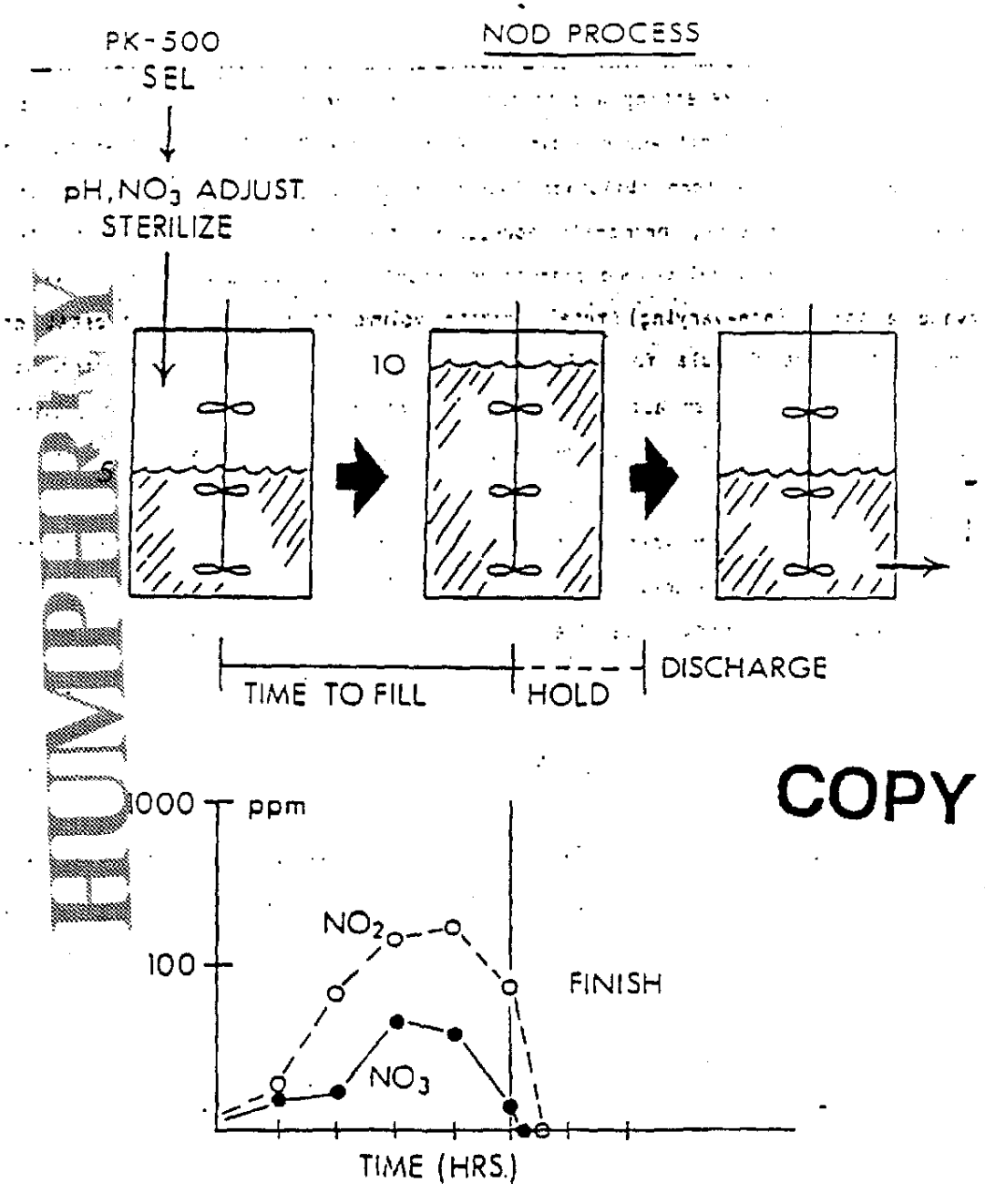

FIGURE 1

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interest was to most accurately describe our working system. Although this would best be done by integrating these changes in the actual working volume with time, we decided to approximate this by simply using the arithmetic mean filling (working) volume half-way through fill as "v". We rationalize this because we know that "the system" doing the work only attains it's true total working volume when it is finished filling, and in a majority of instances, the work has been completed (or nearly completed) by the end of the fill. Thus, some constantly changing volume (dv/dt) that is almost always less than the final full volume is doing almost all of the work. This is diagramatically represented in Figure I.

Furthermore, we also know that in other instances, the total cycle time can be longer than just the total filling time (our process isn't always finished exactly when the fermentor is finished filling, thereby requiring a "post fill" finishing period). Thus, we also could not simply use the value for the rate of addition of substrate per hour ("f") either. Instead, we settled on the following formulation:

$$\frac{f_t + T_t}{V_{ave}}$$

where f_t is the total volume of substrate added to the fermentor during that fill (e.g., the replacement volume); T_t is the total time for that cycle (fill time + finishing time; if not "finished" by the end of fill), and V_{ave} is the mean full (filling) volume of the fermentor (e.g., volume of the "heel" + half the replacement volume). If the time required to discharge the product were substantial, it too would have to be included in the value for the total cycle time. However, since our laboratory-scale fermentors can be discharged in just a few minutes, we have not included this value in our calculations for "D".

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2. Process Chemistry:

Since we have recently documented in detail both 1) our experience over the past 6 months running the mixed culture in shaker-flasks and laboratory-scale fermentors,[13,14] as well as 2) what we currently understand to be the major operational, process and monitoring parameters of the mixed culture denitrification system[15] (Appendix A, attached), we will outline only the relevant data and observations here. Otherwise, any additional information regarding the specifics of start up and shut down procedures, etc. may be obtained from either the aforementioned or following referenced materials.

One, or possibly more of the bacterial strains in our mixed culture mediates the complete dissimilatory removal of Nitrate-Nitrogen to Nitrogenous gas. The process is thermotolerant and anaerobic. Although some question still persists concerning both the positive and negative influences of micro-quantities of oxygen during the initial start-up (induction) period and throughout normal process operation, no extraordinary steps are taken at this time to exclude oxygen from this system. It will probably be some time (months) before this particular point is more adequately understood. Independent of this, the mixed culture appears to be able to cope with rather wide variations in the "quality" of its chemical and physical environment, as demonstrated by its ability (within limits) to grow in and actively denitrify our Park-500 SELs. Recent experiments with single component SELs produced in the R&D Pilot Plant[16] have helped us characterize one of these substrate quality "end points" with regard to the

13. Uydess, I. L., Memo to J. Banyasz, October 7, 1982.
14. Uydess, I. L., Special Report, April 1983.
15. Uydess, I. L., Memo to J. Whidby, March 31, 1983.
16. Thanks to Grant Gellatly and crew.

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acceptability of the major SEL blend constituents. Specifically, 100% Burley, Bright and Oriental lamina SELs were found to be extremely poor substrates for both growth and denitrification, while 100% Burley and Bright stem SELs were each found to be good substrates for growth and denitrification.[17] These observations will be discussed in more detail in a later section. Although we do not as yet fully understand the physiological (biochemical) basis for the adverse impact that each of these lamina SELs had on the performance of our microbial system, the observed effect was significant and reproducible, and helps explain (and predict) how the blend composition of current and future SELs would impact upon our denitrification process. We are currently collaborating with other R&D personnel to elucidate the biochemical basis (with respect to stem and leaf chemistry) for these observations.

Aside from the positive or negative impact of nominal blend composition upon our system, there also appear to be other chemical and/or physical differences within SELs of the same apparent blend composition which effectively render them either "good" or "poor" substrates for NOD. HPLC and other analytical techniques have not, as yet, yielded discernible information on chemical differences between apparently similar substrates (SELs) which could be directly (or reliably) correlated to observed changes in the rate of denitrification (and/or growth), thus "throughput" (D-rate) in these substrates. Previous observations on the consistent disappearance (utilization) of certain organic acids such as malic acid during active growth and denitrification have not been made, per se, thus far in these studies, although our HPLC data do show that there are observable differences between the organic acid profiles of various feed SELs and those of the resultant denitrified products. However, we do not as yet know what these compounds are, nor do we understand how their relative presence or absence

17. NOD Labbook No. 7771.

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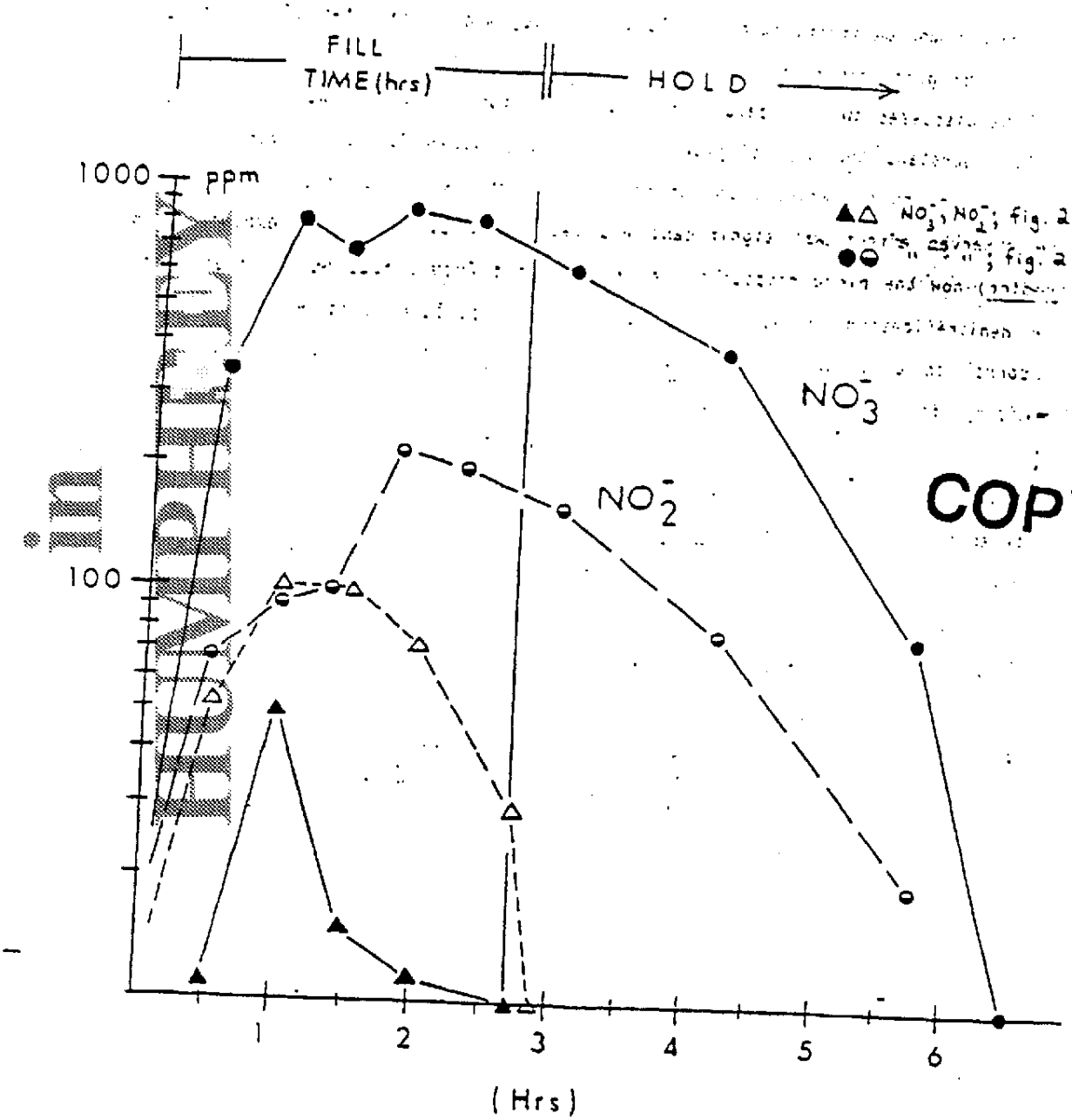
FIG. 2: CYCLE PROFILES



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affect the growth or denitrification capacities of these organisms. There is, however, the documented observation that the relative quality of the SEL does directly influence the rate of denitrification in that SEL.

Normally, when everything is "right," the incoming feed Nitrate is converted to Nitrite at about the same rate at which the feed is added to the fermentor. Therefore, there is normally no appreciable accumulation of residual Nitrate (NO_3^-) during the fill period (e.g., usually >150 ppm). Nitrite, (NO_2^-), on the other hand, usually accumulates to some low-to-moderate residual level during fill (50-250 ppm), but is then converted to gas (N_2O or N_2) by the end of fill, or shortly thereafter (15-30 min). This is best demonstrated by looking at and comparing individual cycle profiles for Nitrate and Nitrite during "good" (fast) and "bad" (slow) cycles. Figure 2A shows a cycle profile for a typical denitrification cycle during which time the Dilution Rate of the fermentor was between 0.15 and 0.20 hrs^{-1}. Figure 2B, in turn, shows another (later) cycle profile for that same fermentor during a period when the D-rate fell below 0.10 hrs^{-1}. As you can see, the phenomenology of Nitrate and Nitrite removal for these cycles is substantially different, and indicative of a functional problem on the part of the microbial system in dealing with either converting Nitrate to Nitrite, Nitrite to gas, or both. Furthermore, it has also been our experience that a transition from "good" to "bad" cycles occurs most frequently at the time of (or shortly following) the introduction of a new batch (new pick-up date) of SEL into the system (e.g., again, indicative of some qualitative difference in our SELs which has an almost immediate impact on the denitrification capacity of the mixed culture). The rapidity with which this adverse impact on denitrification and D-rate is observed is dependent upon several factors including... 1) the previous "health" and stability of the system prior to that SEL's introduction, 2) the actual amount of that material introduced into the system (the

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concentration of new SEL that the system "sees" during feeding of the 10%, 50% or 75% heel), and 3) the magnitude and nature of the problem with that SEL. For now, it would appear that there are several potential problem areas with respect to the "acceptability" of our SELs:

1) Chemical problems: manifested as either insufficient levels of required metabolic substrates to support the growth and denitrification of that bacterial population,
2) An unfavorable physiochemical environment: which inhibits or interferes with transport and/or energy coupling phenomena, such as the presence of inhibitors of bacterial metabolism and/or respiration, or,
3) The presence of alternative metabolic and/or respiratory substrates: which directly or indirectly interfere with the utilization of NO_3^- and/or NO_2^-.

Although it is difficult to say with certainty which of these is the route of most of our problems, it does appear that the lack of appropriate levels of substrate and/or the presence of alternative (competing) substrate are prime candidates.

However, when the environmental conditions for growth and respiration are favorable, the mixed culture can demonstrate an appreciable aptitude for Nitrate and Nitrite removal. Figures 3 and 4 show the individual cycle times and resultant D-rates obtained running the mixed culture in Park 500 SELs with feed Nitrate levels varying between 2200 and 3500 ppm. As you can see, the mixed culture was able to maintain a dilution rate in excess of 0.1 throughout most of this period of varying Nitrate concentrations. However, on other occasions, when unfavorable SELs were introduced into the system (such as 100% Burley lamina

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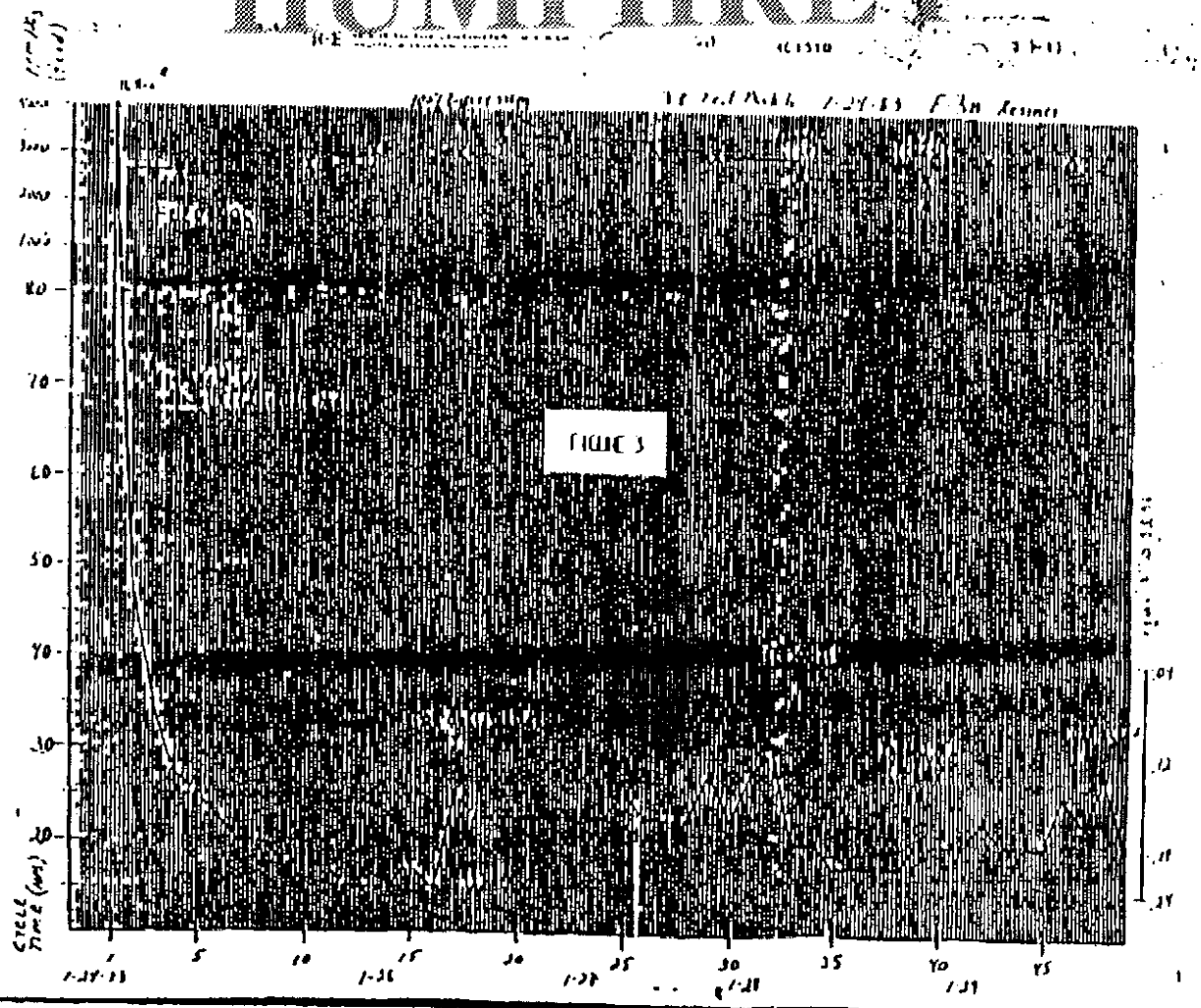

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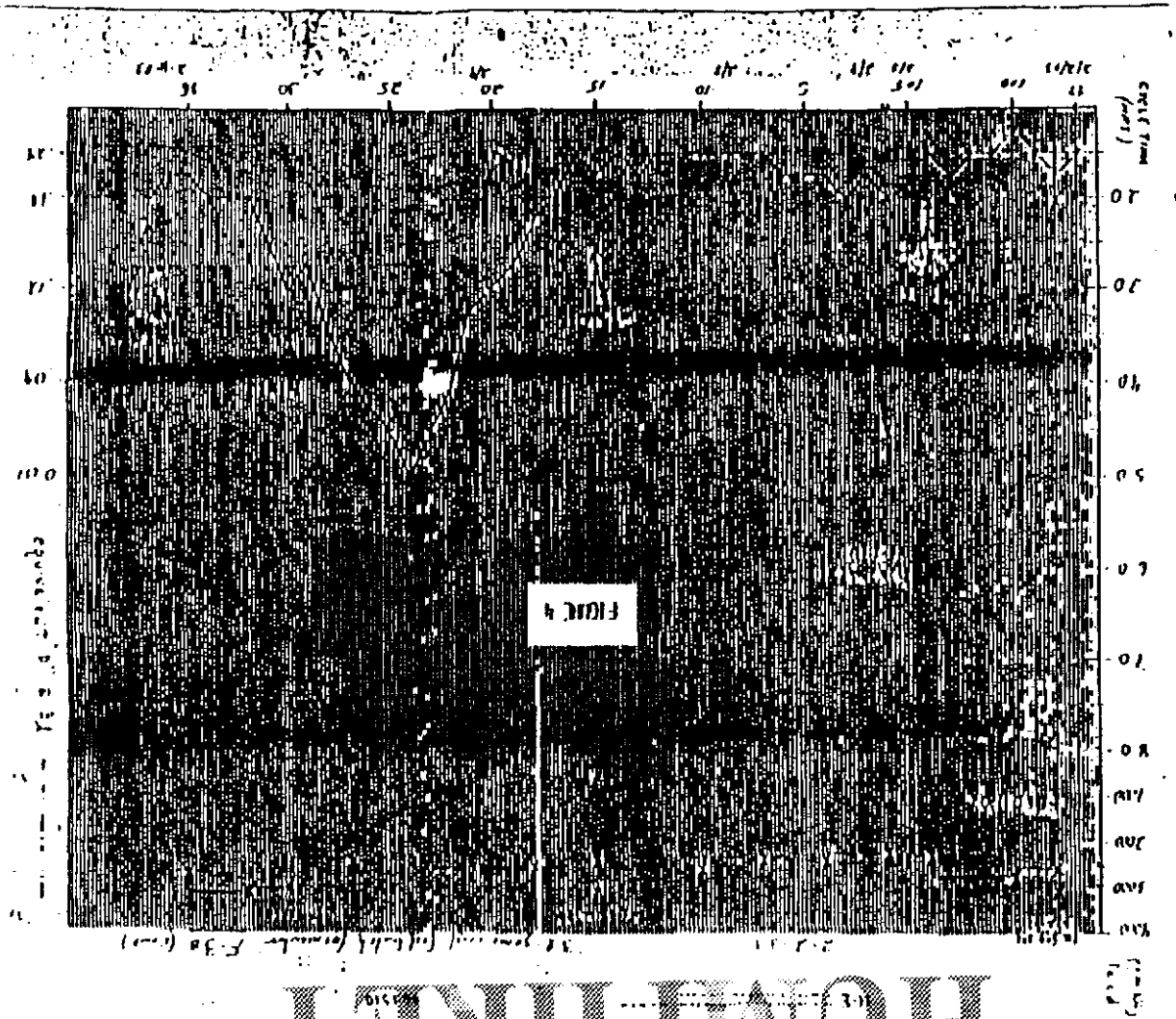

FIGURE 4

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SEL), the O-rate deteriorated sharply soon after its addition to the system (rememoer, the dilution effect).

An understanding for and appreciation of the magnitude of dependency this system has on the quality of its substrate is crucial to attempts to utilize it in an industrial environment. Our SELs do have the potential to vary in chemistry and day-to-day consistency, and this has a direct effect on the throughput of this system irregardless of how large or how small the operating volumes are. We must endeavor to identify those variable SEL components that are responsible for this biological impact so that we may either; 1) optimize SEL production to avoid (or compensate for) these variabilities, or 2) supplement our SELs with these materials (organic acids?). Furthermore, we also have to be able to predict the operational impact that certain SELs will have on our system prior to their introduction into it. This would also necessitate the testing of future blend changes in a laboratory or Pilot Plant situation as support for the operation of such a facility. These unknown variabilities can have an even greater adverse impact when poor quality SELs are inadvertently used during de novo start ups. Having to cope with the adverse impact of an unsuitable environment is one thing when the culture is "up and running," but having to rely upon it during initial start up from stored feed, is quite another.

3. Nitrate and Nitrite Removal:

Spectrophotometric and GC "headspace" analysis of actively denitrifying fermentors have demonstrated that the feed Nitrate (and resultant Nitrite) are completely removed to nitrogenous gas (N_2O and/or N_2). However, the relationship between which specific off-gas (N_2 or N_2O) is the major or sole nitrogenous off-gas product and the O-rate of the operating fermentor (or initial concentration of Nitrate in the feed) is as yet unclear. We have observed on

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numerous occasions that apparently "equivalent" fermentors (operating at the same D-rate) may have significantly different off-gas profiles with respect to the presence and/or ratio of N_2 and N_2O. Aside from N_2O and N_2, the only other off-gas product of significant concentration (>1%) is CO_2, which appears to predominate (>50% of the total off-gases) when the D-rate is poor (<0.1). This is not particularly surprising since one would probably not expect to find much N_2 or N_2O issuing from a fermentor that isn't removing NO_3^- to NO_2^-, or NO_2^- to gas. Whether GC off-gas profiles can be used to monitor the status of the operating system or predict an impending problem, has yet to be seen. More specific information concerning off-gas profiles for actively denitrifying fermentors, as well as the determination of the specific Heat of Reaction for Nitrate conversion to Nitrite, and Nitrite to gas, can be obtained from the references of Watson, D., as well as from that of Martin, P. and Counts, M. E.

4. HPLC and Analytical Data:

Although previous studies[13] reported that Malic Acid was frequently consumed by the mixed culture system during periods of active growth and denitrification, recent HPLC determinations of sterile PK-500 feeds and their subsequent denitrified products have not proven this to be characteristic of the current system. However, we have observed significant fluctuations in the various (including organic acids) peak heights of our pre- and post fermentation HPLC profiles. These encompass both increases and decreases of pre-existing (feed profile) peaks, as well as the appearance of new peaks, presumably the result of the metabolic conversion or degradation of other materials within our feed SELs. Although we have recently increased the spectrum of our known standards (to include Acetic, Propionic, Butyric, Malic, Lactic, etc.), we have not, as yet,

13. See Banyasz, J., Proj. 1904 Annual Report, May 21, 1982, Acc. No. 82-159.

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been able to make any meaningful correlations between the efficacy of denitrification (D-rate) and these pre and post denitrification organic acid profiles. In light of the mixed culture nature of our system, the observed fluctuations in organic acids could easily reflect the complex nature and interrelationship of the various microbial metabolisms being expressed within an equally complex and ill-defined substrate, our SEL. Some of these plant-derived materials in our SELs are most certainly principal metabolic substrates for one or several of these bacteria. However, other microorganisms within the mixed population may well be producing more of these same (and/or different) materials as they metabolize their specific substrates. Hopefully, we will be able to elucidate more about this in the near future as more is learned about the physiologies of the individual participants in this mixed culture of microorganisms.

Data generated in collaboration with the Analytical Division has helped verify the following with regard to the ranges of conditions within which the mixed culture will continue to grow and denitrify in actively denitrifying fermentors:

a. Operating pH:

The "running" pH of actively denitrifying fermentors has been shown to be dependent upon 1) the blend composition of the feed SEL (with respect to its "as-is" pH and buffering capacity), and 2) the original Nitrate level in the feed. The higher the original feed Nitrate level, the higher the resultant "running" and "finishing" pH of the actively denitrifying fermentor. pH with respect to blend composition, is primarily determined by the relative quantities of Bright and/or Burley lamina, and the relative ratios of lamina vs. stem. This relationship between SEL pH and blend composition is known, and has been documented previously (G. Bokelman, 1982). However, there are still certain aspects of the chemistries (buffering capacities, etc.) of our SELs which are

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still unknown, which do appear to have a significant impact upon the mixed culture's ability to grow and/or denitrify within these materials. A better understanding of the nature of this variability in SEL chemistry is paramount to our being able to predict and possibly compensate for how well some SELs will run in our mixed culture denitrification system.

b. SEL Total Solids:

Experiments were conducted to determine if the mixed culture could continue to grow and actively denitrify within SELs having elevated solids levels. To accomplish this, Pk-500 CEL was added back to standard Pk-500 SEL to achieve a total solids level in excess of 15%. These high solids (and high Nitrate) SELs were then fed to an actively denitrifying fermentor running up to that point on 7-8% solids (Pk-500 SEL) feeds. Although the initial addition(s) of this high solids feed to the 50% heel of the fermentor diluted it somewhat, successive 50% volume additions soon elevated the "working" solids level within the fermentor to approximately 14%. The Nitrate levels of these CEL-fortified SELs was also high, and in the range of 3600 to 4000 ppm. The mixed culture continued to grow and actively denitrify these high solids/high NO_3^- SELs up to and including a level of 14% at dilution rates in excess of 10%. However, it is not known whether this can be accomplished within all composition SELs, nor do we know at this point what the longevity of this denitrification capacity would be within such high solids SELs during long term operation. The total alkaloids levels within these high solids SEL feeds were also elevated, and were found to be about 3.0 - 3.5% in the resultant denitrified products.

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5. FEED SEL NITRITE:

Investigations were also conducted to determine the effect of elevated feed Nitrite (NO_2^-) levels on the operational consistency of an actively denitrifying fermentor. To do this, Potassium Nitrite (KNO_2) was added in successively higher concentrations to our SEL feeds prior to sterilization to obtain Feed NO_2^- levels between 200 and 1000 ppm. These Nitrite-fortified SELs were then used to feed an actively denitrifying fermentor. Within forty-eight hours, SEL feeds with Nitrite levels in excess of 600-800 ppm were being denitrified without any long term perturbations to the throughput of the fermentor (which continued to operate in excess of a 10% dilution rate).

6. SEL BLEND COMPOSITION:

One of our foremost project goals was to determine the effect of changes in blend composition on the denitrification capacity of the mixed culture. With the assistance of Grant Gelatly and the R&D Pilot Plant crew, 100% Burley, Bright and Oriental lamina SELs were produced, together with 100% Bright and Burley stem SELs. Each was then diluted to a 7-8% solids level, fortified with KNO_3 to ≈ 2500 ppms, and introduced into actively-denitrifying fermentors. As mentioned previously, the 100% stem SELs (Burley and Bright) ran well, while the 100% lamina SELs (Burley, Bright and Oriental) did not. In fact, the successive addition of the 100% lamina SELs to the actively denitrifying fermentors resulted in a more or less immediate deterioration of the dilution rate from well above 10% to well below 10%. The mixed culture denitrification system does not appear to be able to handle 100% lamina SELs. Mixtures of these 100% lamina and stem SELs will be examined in the near future to determine end points for the maximum % lamina content in SEL that this microbial system can run. Such experiments as these would be useful in determining the impact of future blend compositions changes on the NOO system running at Park-500.

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7. PRE-ADJUSTMENT OF FEED SELs:

Although it has previously been demonstrated that this system requires no exogenous additives, recent experience has shown that a slight upward adjustment of the pH of our sterilized feed SELs generally promotes more consistent process operation and better throughput (all other factors being held equal). As a result, 1 1/2 to 3 mls of 30% Ammonium Hydroxide are currently being added to our sterilized SEL feeds to raise their post-autoclaved pH from about 5.2 to approximately 6.0. Alternative bases such as KOH and DAP have also been used in a similar manner with equal success, although the prolonged use of DAP-adjusted feeds did result in the build-up of large quantities of a brittle "scale" on the inside of the process fermentors necessitating frequent cleaning - thus, interruption of the processes.

Experiments conducted in the winter of 1982 using '82 blend composition Pk-500 SEL demonstrated that higher D-rates could usually be achieved by supplementing low NO_3^- Feed SELs (1400-1300 ppm) with additional quantities of nitrate, elevating such feeds to a ±2500 ppm (+/- 300 ppm) level using KNO_3 resulted in higher and more consistent D-rates. However, we currently find that the mixed culture system runs equally as well on '83 blend Pk-500 SELs having "as-is" nitrate levels anywhere between ±1400-2400 ppm. Higher nitrate levels (>3000 ppm) may or may not run better, but we now know this to be highly dependent upon the chemical quality of our SELs as metabolic substrates for the mixed culture [illegible] with 3-5000 ppms NO_3^- have been run at dilution rates in excess of [illegible]%).

8. START-UP AND SHUT-DOWN PROCEDURES:

The specific details of our current operating process including start-up and shut-down procedures are given in our March 31, 1983 memo to

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Dr. J. Whidby, appended herein (Appendix A). Recent experiences with 10% - 30% inoculum "batch" start-ups have taken us a step further by demonstrating (to us) how critically-dependent such de novo batch start-ups are upon 1) the exact level of inoculum used (thus, number of viable microorganisms within), 2) it's past history with regard to age and "condition," and 3) the temperature of the start-up. It now appears that although "older" seed (>2-3 months old) will start up, (albeit, slowly), newer seed is usually more reliable in that it invariably starts up faster. Furthermore, independent of the relative quality of the stored seed, maximization of the percent (viable) inoculum used at initial start-up maximizes the chances of success. It is now evident that successful (reliable) short-term start-ups (<24 hrs) from refrigerated seed are highly dependent upon the presence of a critical minimal population of viable and "competent" microorganism(s) upon which the functionality of the entire microbial population depends under the unique environmental pressures of a low level inoculum (10-30%) batch start-up in chemically-variable SELs. We do not as yet fully understand the basis for these observations, but we do hope to be able to more fully elucidate these phenomena when more is known about the metabolisms and proportions of the individual participants within our mixed culture. Additionally, we now find cold (13°C) de novo batch start-ups to be generally more efficacious than those previously implemented at 47°C. Currently, refrigerated seed (stored at ≈4°C) is allowed to warm at room temperature to 14-18°C and is then added as a 30% inoculum to a sterile fermentor containing 7 ls of cooled (≈16-18°C) sterile SEL, pH 6.8 to 7.2, 1800-2200 ppm NO_3^-. The optimization of consistent and reliable de novo start-ups from inactive, stored seed still remains one of our chief goals. We are currently exploring alternative techniques and procedures for attaining this goal.

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9. PRODUCTION OF SEED FOR THE R&D PILOT PLANT START-UP:

In March 1983, start-up inocula (seed) was produced in the laboratory from Pilot Plant SEL, the composition of which was generally modeled after the 1982-83 Pk-500 SEL blend compositions. Approximately 80-100 liters of seed were collected aseptically in 2 l aliquots and stored at 2-4°C. Several samples of this seed have recently been used as 30% inocula to successfully start up fermentors in the laboratory.

10. CHARACTERIZATION OF THE MIXED CULTURE:

Experiments are currently under way in collaboration with Dr. Walter P. Hempfling, University of Rochester, NY, to elucidate the identities and capacities of each of the microorganisms within the mixed culture. This information will be used to further optimize this process in the future.

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CONCLUSIONS AND SUMMARY:

1. The mixed culture of tobacco-identical microorganisms has been shown to be a stable and potent mediator of the thermotolerant, anaerobic dissimilatory denitrification of our Pk-300 SELs.

2. Industrially acceptable dilution rates in excess of 10% volumes/hr have been demonstrated over prolonged periods of operation in the semi-continuous mode over a wide range of SEL conditions including:
   a. Nitrate-N levels between 1400 and 3200 ppm.
   b. Feed Nitrite levels up to 800 ppm.
   c. Total solids levels to 14%, and
   d. Total alkaloids to 3.0%.

3. The reliability of process operation, temporary hold, shut-down and re-start (re-activation) procedures has been improved substantially as a result of an increased understanding of the impact of substrate (Nitrate) and environment (temperature) upon the mixed culture.

4. We continue to investigate the phenomenological basis for both problematic and efficacious de novo start-ups from refrigerated seed. Progress has been made, within the limitations of our current understanding of this process, to improve start-up reliability by 1) increasing the initial inoculum used to 30%, and by 2) avoiding sharp thermal transitions (shock) at the time of inoculation by employing a cold, 13°C start-up procedure similar to our laboratory short-term operational shut-down and start-up procedures. Alternative procedures are also being investigated.

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5. Experiments on the impact of individual (100%) SEL blend components on NOD have demonstrated that 100% lamina SELs are not acceptable substrates for the current mixed culture denitrification process, but that 100% stem SELs are.

6. Collaborative studies with a wide variety of personnel within PM R&D and Engineering have elucidated numerous process and operating parameters important to both our own understanding (and improvement) of our process, as well as to the design and engineering of the R&D Pilot Plant and Park-500 NOD facilities.

7. Certified seed has been made (and tested) for the Pilot Plant start-up in May, 1983.

8. Antibody to ATCC-1 has been made and tested. The resultant immunofluorescent tests demonstrate the presence of organisms similar in antigenicity to ATCC-1 in the mixed culture.

9. Evaluations of laboratory NOD product are currently under way to characterize their subjectives.

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ADDENDUM:

Although we are far from mastering an understanding of the intricacies of this microbially-mediated denitrification process; we have, with the help of an ever-growing community of collaborators, been able to substantially improve our appreciation for the strengths and weaknesses of our current NDD process. This has recently led (directly, or not) to advances in the reliability and consistency of our process. We are confident that continued studies in these areas will even further enhance our ability to understand and utilize this and future biotechnology processes to make commercially-successful products here at Philip Morris.

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ACKNOWLEDGMENTS AND PERSONNEL:

The progress made to date in our understanding of the laboratory-scaled MCO mixed culture system is largely due to the extensive efforts of each of the personel within Project 1904, many of whom worked unusually long hours over extended periods of time to keep our long-term semicontinuous fermentation experiments going. Special thanks are extended to Robert Dunn for his diligence and efficiency in helping schedule both personnel and experiments for the numerous interrelated and overlapping investigations that have been conducted over these past six months.

Project 1904 personnel would also like to acknowledge their appreciation for the extensive collaborative efforts of a variety of other researchers and facilities within PM R&D, including; Duane Watson, et al., (GC); Betty Handy and Bill Harvey, et al., (Analytical); Grant Gellatly, et al., (Pilot Plant CELs); Bob McEuen and Edie Taylor (Immunofluorescence); and Ray Harding and other Pk-500 personnel (SELs).

We would also like to thank our PM management and scientists for their continued support (incl., William A. Farone, Jerry Whidby, Cliff Lilly and Ted Mansell) and our consultants for their valuable suggestions and guidance, Walter P. Hempfling (University of Rochester, NY), and William Stark (PEDCo).

Project 1904 Personnel are: J. Banyasz[1], P. Kusel, R. Dunn, D. Chadick, S. Tenhet, P. Oglesby, E. Bravo, G. Fortner[2], P. Boone, G. Nixon[1], A. Conyers, P. Finn, M. Varnier and I. L. Uydess.

1. Currently in other projects.
2. No longer with PM R&D.

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INTER-OFFICE CORRESPONDENCE

RICHMOND, VIRGINIA

To: J. F. Whidby Date: March 31, 1983

From: I. L. Uydess

Subject: Operational and Process Parameters of the Mixed Culture Denitrification System

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I. DE NOVO START-UP FROM STORED, REFRIGERATED SEED

Definition of:

A. SEED: Start-up inoculum consists of 1-2 liters of refrigerated product from previous successful denitrification run(s) using Park 500 SEL as feed. Product from said run (cycle) is collected in a sterilized polypropylene (polyethylene) 2-liter, screw cap storage jar and placed immediately into the cold room after being labeled with the following information:

- I.D. Number of Fermentor Vessel of Origin
- Denitrification Run (Cycle Number) From Which Seed Was Collected
- Date and Time of Collection
- ppm's NO_3 and NO_2 in Product
- pH of Product
- Presence or Absence of Butyric Acid (Normally Never Present in Product)

Additional data of import regarding the overall "quality" of the product (and potential inoculum) that is recorded in lab notebook(s):

- Cycle Time of That (and Every) Run
- Extent of NO_3 and NO_2 Accumulation During Run (Cycle Prophile)
- Initial and Final pH of Run
- Nitrate Level of Feed Used in That Run
- Cell Number in Fermentor (Thus Product)
- HPLC Organic Acids Data (Acetic, Propionic, Butyric, others)
- Off-Gas Profiles Typical of Collection Period

Normally, only product from successful runs is collected for storage as seed. This means:

- That the cycle time at the time of collection of the product (seed) was at least 0.1 "D" or better (typically, a dilution rate of 15%).
- That NO_3 and NO_2 are N.D. in the product (NO_2 is typically 50-100 ppm at the time of collection but goes to zero immediately afterwards).
- That there is no butyric acid in the product (or in the fermentor producing the product).
- That the phenomenology of NO_3 and NO_2 removal during that run was "normal"--e.g., that there were no unusual or significantly high

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accumulations of either NO_3 or NO_2 during the run. (Otherwise, this would be indicative of a problem in removing NO_3 and/or NO_2 during that cycle).

- That the product is from the denitrification of a "standard" SEL--not a specialized SEL formulation being used for experimentation.

- That the nitrate in the feed SEL being denitrified at the time of collection of the product (seed) was within the normal recommended range (approximately 2200-2800 ppm NO_3).

- That the feed SEL used during the collection of that seed had no "off" characteristics--such as: 1) the presence of "out-of-spec" quantities or organic acids (proprionic or, especially, butyric), 2) >100 ppm NO_2, and/or 3) an unusually high initial pH that is not known to be due to a change in blend composition, but rather, to be indicative of prior microbial activity in the feed.

B. FEED: Acceptable feedstock for mixed culture denitrification has been defined as SEL product from the PM Park 500 facility which has the following "as-is" characteristics:

- 1200 to 3200 ppm's NO_3 (depending upon blend composition and line of origin).

- Butyric Acid - None (unless this is known to be due to blend components used and not to prior microbial activity).

- Propionic Acid - None (unless this is also known to be due to blend components used and not to prior microbial activity).

- Acetic Acid no higher than 3-4,000 ppm.

- "As-is" pH of 5.3 to 5.8. (This depends upon SEL blend composition and may vary slightly due to the presence or absence of higher or lower proportions of Burley vs. Bright, stem vs. lamina, etc.) *However, unusually high pH can also be indicative of prior microbial activity, but this is usually accompanied by unexpectedly low NO_3 levels and high NO_2 levels and may also be accompanied by higher than normal concentrations of proprionic and/or butyric acids.

- Post-sterilization (post-autoclaving) pH of 4.9 to 5.4. Again, this is somewhat dependent upon the original "as-is" pH and can be blend dependent. However, unusually low pH's (lower than pH 4.9) have, in the past, been indicative of possible off-spec.-chemistry SEL (high concentration of Bright lamina?) which are more difficult to run and often result in longer cycle times, lower "D" rates, and poor operational consistency.

- Soluble Solids: nominally 6-10% (5-15% have been run).

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. Insoluble Solids: (We have not tested this yet.)

. Total Solids: nominally 6-10%.

. Total Alkaloids: nominally 0.1-0.3%.

In-house Adjustment of Feed:

Purpose: In order to increase the efficiency and optimize the consistency of the mixed culture denitrification process, the following guidelines have been adopted:

. Increase NO_3 (if necessary) to 2500 ppm +/- 300 ppm by the addition of KNO_3 prior to sterilization.

. pH adjustment of the post-sterilized feed SEL to between pH 5.8 and 6.0 by the addition of 30% NH_4OH.

C. De novo START-UP: When a complete restart of process is required necessitating the utilization of a small quantity of stored, refrigerated seed which must be amplified by serial (1:10) increases in volume.

1. Fed Start-up:

. One or two liters of stored inoculum is transferred aseptically into a sterile, 14-liter fermentor vessel already on a fermentor mainframe and hooked up to a sterile, preadjusted feed (approximately 2500 ppm NO_3, pH 5.8-6.0). Alternatively, the seed could be added to a vessel that already contained 1-2 equivalent volumes of sterile, preadjusted feed. In this instance, this initial volume of feed may have to be denitrified before feeding of additional SEL is begun.

. Feeding is then initiated at approximately 0.1 volumes/hr (10% volume per hour) using 47°C preadjusted feed.

. NO_3, NO_2 and pH are monitored periodically throughout the fill (usually at each 0.1 volumes of addition--e.g. at each 1 liter of addition of feed to a final working volume of 10 liters in the 14-liter fermentor). The fermentor itself is set to operate at 47°C.

. If, during the initial adjustment period (of increasing temperatures and the addition of fresh feed) NO_3 and/or NO_2 levels build to greater than 100-150 ppm NO_3 and/or >300-400 ppm NO_2, feeding is temporarily discontinued until NO_3 goes to N.D. and NO_2 decreases to ≤100 ppm. Feeding is then resumed at 0.1 "D".

. Monitoring of NO_3, NO_2 and pH should be continued (at each addition of 1 liter during fill) to verify efficacy of start-up.

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. When final working volume has been reached (10 liters), and the reactor is verified to be N.D. NO_3 and <100 ppm NO_2, one-half (1/2) of that total volume (e.g. 5 liters) is pumped off rapidly until a 5-liter heel remains. Discharge of product is then discontinued, and feeding (at 0.1, 0.15, or whatever feed rate is required by system) is reinitiated until the final 10-liter volume is attained again.

. NO_3 and NO_2 (as well as pH) should be monitored each 0.1 volumes during fill to again verify the operational status of the system (that it is running at ≤100 ppm NO_3 and ≤250 ppm NO_2 during fill--otherwise, an adjustment in feed rate may be required).

. If NO_3 and/or NO_2 are not approximately N.D. (N.D. NO_3 and ≤100 ppm NO_2) at end of fill, a variable post-fill "finishing" period may be required. This can be normal, and often depends upon (and reflects) the "quality" of the feed SEL and operational status of the mixed culture. However, when the process is running at a "D" rate of 0.1 or better (0.15, etc.), such a finishing period should not exceed 10-30% of the fill time (depending upon the length of the fill time).

. Typically, in a 5-liter heel system, the fill time should be approximately 3-3.5 hours, and denitrification should be completed either when the fermentor is full, or within 30 minutes to one hour thereafter.

2. STRAIGHT BATCH START-UP: Alternatively, the NOD process can also be started from stored, refrigerated seed using a 1:10 batch start-up.

One liter of refrigerated seed is transfered aseptically into a sterile, agitated 14-liter fermentor containing 9 liters of preadjusted SEL feed held at 47°C.

NO_3, NO_2 and pH are then monitored at 1-2 hour intervals until denitrification of the initial 10-liter volume of feed (feed plus seed) is verified.

Fifty percent of that fermentor volume is then discharged rapidly and the addition of fresh feed begun at approximately 0.1 volumes (10%) per hour until that fermentor is full once more.

. NO_3, NO_2 and pH are monitored at each 0.1 volumes of addition (in this case, at each one liter of addition) to ascertain the operational status of that fill.

. If denitrification is not completed by the end of the fill interval, a variable post-fill finishing period may be required. NO_3, NO_2 and pH samples are taken as needed during this interval until denitrification is complete. However, as stated before, this period should not exceed 20-30% of the fill time, depending

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upon 1) the actual duration of that fill time, 2) the feed NO_3 concentration, and 3) the previous "O" rate of that system).

. The interval required for the complete denitrification of the initial 10-liter volume of feed is typically between 15 and 30 hours, depending upon the "quality" of both the feed and the seed.

II. PROCESS OPERATION: (Assuming a 50% Heel)

A. Shutdown: In the event of a planned shutdown for greater than 12 hours (typically, for one day to two weeks), during which time 25% or more of the maximum fermentor capacity would be held and maintained at 4°C (in the fermentor) to be used as inoculum for a subsequent run:

. On the last planned cycle, slow fill the first half of the fill volume at the nominal fill rate, then batch in the last half of the fill volume. Stop heating; cool to 4°C. Stop agitation; maintain at approximately 4°C.

B. Start-up: (from refrigerated full or partially full fermentor containing "held" product):

. Discontinue cooling, resume agitation and when fully mixed, discharge 50% of contents. Begin heating to 50°C. When fermentor temperature reaches 20-25°C, resume feeding (initially at about 10% per hour) until full. When NO_3 and NO_2 are zero, discharge product and resume normal fill cycle (normal operation).

To Be Monitored While Feeding During Initial Start-up Cycle): NO_3 and NO_2 levels in fermentor must be checked--usually as each additional 10% of volume is added to determine status of the mixed culture (e.g., are NO_3 and NO_2 being consumed normally, or not?). Usually, bacteria will become active (with respect to their ability to remove NO_3 and NO_2) above 25°C. If they are "healthy" (if previous shutdown did not perturb culture and, if current SEL is acceptable), the mixed culture will consume incoming NO_3 and NO_2 during warm-up. Organic acids (acetic, proprionic and butyric) must also be monitored to verify status of "held" (refrigerated) culture. Normally, some proprionic acid (200-300 ppm) may be found to have accumulated during cool-down and/or storage. This could be due to metabolic changes resulting from the progressive temperature shifts experienced by the culture, or, because of alternative metabolisms be expressed if NO_3 and NO_2 are depleted during cool-down before the mixed culture becomes metabolically inactive. No butyric acid should be present in the start-up mixture. Microscopic cell counts and viable plating should also be done to verify cell number.

C. TEMPORARY HOLDS (SCHEDULED, OR OTHERWISE):

When a temporary disruption in the operational status of the MOD facility precludes either A) the discharge of a finished volume of product, or, B) refeeding of the saved heel (e.g., starting a new cycle), the following procedure can be employed:

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. "X" volumes of a concentrated, sterile solution of KNO_3 may be added (batch or fed) to the full or half-full fermentor containing the denitrified product so as to raise the NO_3 level in that product to between 1000 and 2000 ppm.

. NO_3, NO_2 and pH are then monitored at 1/2 to 1 hour intervals until all of the added NO_3 (and resultant NO_2) is removed. The length of time that this denitrification cycle would take is predictable, and would be related to 1) the prior NO_3-removing capacity (history) of that fermentor, 2) the amount of NO_3 added, 3) the extent of the microbial population, and 4) the "quality" of that fixed volume of SEL (with particular regard to the persistence of excess substrates remaining in that volume of previously denitrified SEL).

. Once this additional NO_3 has been removed, normal process operation should be resumed.

. The efficacy of this short-term (one cycle) holding procedure is limited and highly dependent upon the substrate quality of the original feed SEL.

D. NOMINAL PROCESS OPERATION:

Once the system is up and running, a "D" rate of 0.1 $hours^{-1}$ should be attained within 2-3 cycles after start-up (if no complications arise due to either problems experienced prior to or during shutdown or start-up). If all is well during shutdown and start-up, the initial cycle of start-up should take no longer than 8-12 hours, assuming a 50% refrigerated heel, 10% feed rate, and the normal delay in microbial activity experienced as a result of the temperature shift from 4°C to 47°C. Each subsequent cycle should then be 50 to 75% the time of the previous cycle until the nominal 0.1 "D" rate of 5 hours is reached (again, this is normally attained within 3-4 cycles, including the initial, cold, start-up cycle). Worst case thus far, 25 hours to attain ≥0.1 "D"; and best case, 7 hours to attain ≥0.1 "D". It is normally 8 to 12 hours.

E. NOMINAL PROCESS MONITORING: When working with a new (as of yet, unfamiliar) system.

. NO_3 and NO_2 should be checked at each 10% volume level increase during fill and at required intervals afterwards if product is not completely denitrified by the time of completion of fill.

. pH should be monitored during fill and especially during final interval of denitrification (nitrite removal) to verify "normal" phenomenology of cycle (e.g., that the pH increases as NO_2 goes to N.D., and also, that the final pH at the time of completion of the run is about the same as that of previous runs). However, this is also somewhat dependent upon normal day-to-day variations in the overall quality of the Park 500 SEL with regard to: 1) chemistry of the incoming feed (blend and/or other

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compositional changes affecting buffering capacity, etc., and 2) initial NO_3 level in the feed (the higher the feed SEL NO_3 level, the higher the resultant finishing pH).

Other parameters to be monitored:

. Cell Number: once per shift or whenever a change occurs.

. Organic Acids (HPLC): once per shift or whenever a change occurs.

. Off-Gases (GC): periodically or whenever a change occurs.

. Colony Morphology (Plating): interval varies (daily).

B. PROCESS MONITORING: (Working with a known system.)

Once some familiarity (experience) with the system has been acquired:

. NO_3 and NO_2 should be checked at end of the fill cycle and, if required, subsequently at the completion of denitrification.

. pH at end of fill cycle (and, if required, subsequently at completion of denitrification).

. Cell Number: once per shift and whenever a change occurs.

. Acids: once per shift for both the feed and fermentor(s) or whenever a change occurs.

III. WHAT WE KNOW ABOUT "GOOD" AND "BAD" CYCLES:

A. Characteristics of "Good" Cycles:

. "D" rate $\geq$ 0.1 hrs^{-1} (typically 0.15 hrs^{-1} or 15%).

. NO_3 never accumulates to more than 50-100 ppm during fill (usually by mid fill and $\leq$50-100 to N.D. by end of fill).

. NO_2 does not accumulate to more than 350 ppm during fill (typically 50-250 ppm by end of fill) and goes to N.D. within 30-45 minutes after fill.

. pH decreases slightly during initial period of fill (due to addition of acidic SEL feed), but increases during last half of fill (and/or finishing) due to the removal of NO_2.

. pH at end of fill approximately same as "finishing pH" of previous good cycles.

. No butyric acid produced.

. No propionic acid produced.

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. Cell number $\geq$ $1x10^9$ cells/ml (typically 2 to $4x10^9$ cells/ml).

. N_2 and N_2O major off-gas products (as opposed to CO_2).

3. Characteristics of a Problematic Cycle:

. "O" rate <0.1 hrs^{-1} (or an abrupt decrease in "O" rate from previous cycle - e.g., $\geq$50% of that previous "O").

. NO_3 accumulates to more than 150 ppm during fill.

. NO_3 present (to over 150 ppm) after completion of fill.

. NO_2 $\geq$350 ppm during fill.

. NO_2 increases to greater than 300 after completion of fill (as the residual NO_3 goes to NO_2).

. Time to finish after fill is more than 1/2 of total fill cycle.

. pH remains low throughout fill.

. pH at end of fill and/or end of finishing is significantly lower than "normal" finishing pH of previous runs unless this is known to be due to a blend or feed nitrate level change.

. Some butyric acid produced (if present, indicative of problem possibly related to "quality" of feed).

. Propionic (although this is variable, and 200-300 ppm's are not uncommon during periods of successive, slow runs).

. Cell number of <$1x10^9$ (or abrupt decrease in cell number). Cell number - (total cell count or viable count) may not decrease if slow cycle time is related to a transitory respiratory or metabolic problem that manifests itself in slow NO_3 and NO_2 removal only. However, slow cycle times accompanied by a significant decrease in cell number is indicative of a much more significant microbiological problem affecting cell growth.

. CO_2 major off-gas (proportion of CO_2 to N_2+N_2O increase).

/lvb

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INTER-OFFICE CORRESPONDENCE

RICHMOND, VIRGINIA

APR 29 1983

J. F. KNIGHT

to: Distribution

date: April 27, 1983

from: K. H. Cho

subject: PROJECT CANCELLATION - H.O.D. PLANT - PARK 500
PROJECT NUMBER BC26-0002

This is to notification that H.O.D. Plant - Park 500 project (Project Number BC26-0002) has been cancelled effective April 26, 1983.

Pedco Inc. has been notified to stop all activities for this project. Project closing work with Pedco Inc. is in process.

All project documents will be filed in Central File at the Operations Center. Each member of the project team may keep his own file in his own way. If you prefer to turn over to central file, please hand me the files by May 5, 1983.

Although it was a short time, I appreciate your cooperation and it was a pleasure working with you.

K. H. Cho

/pa

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PHILIP MORRIS U. S. A.
INTER-OFFICE CORRESPONDENCE
RICHMOND, VIRGINIA

RECEIVED
MAR 24 1982
PHILIP MORRIS

To: Mr. H. G. Burnley, Jr. Date: March 19, 1982

From: J. C. Gomes

Subject: STATUS REPORT - M.O.O. PROJECT
PROJECT NUMBER 8C26-0001

Attached for your information and review is a report on the status of the M.O.O. project. It consists of:

I. OBJECTIVE
II. CONCLUSIONS
III. PLANS
IV. DISCUSSION

J. C. Gomes

/bvb

Attachment

cc: Mr. A. S. Church
Mr. J. F. Whidby
Mr. J. A. Williams
Central File 8C26-0001

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SUBJECT: N.O.D. PROJECT STATUS

I. OBJECTIVE

Present an overview status of the N.O.D. project for management review.

II. CONCLUSIONS

1. Denitration by bacterial fermentation has been achieved in the pilot plant, both by fed-batch and continuous mode operation.

2. Subjective indications of product quality and acceptability are favorable and official evaluations are in progress. However, quality and acceptability of product denitration by bacterial fermentation has not yet been approved.

3. Consistent denitration rate results have not yet been achieved in the pilot facility. This is primarily attributed to variations in feedstock composition (i.e., solids, nitrates, nutrients, inhibitors, etc.) and excessive levels of contaminating organisms.

4. Successful process startup has been repeated in the lab several times, but only once in the pilot plant. This was done using pH adjustment and inoculum grown and harvested in sterile SEL.

5. Sterilization of the entire process feedstream will be required to control contaminants which produce unacceptable levels of organic acids. Primarily butyric acid.

6. Excessive levels of organic acids are unacceptable because of the odor and taste they introduce to the product. Subjective testing is in progress on sheet runs made with SEL denitrated in the pilot plant during February 1982, to determine acceptable acid levels.

7. Pilot plant modifications will be required to sterilize and maintain levels of contamination acceptable to this process.

8. The known process conditions at this time are pH (between 7 and 8.5) and temperature (50°C ± 2).

9. The nutritional requirements and growth inhibitors for the desirable organism are not yet completely known.

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10. The impact of variations in feedstock composition on the process are not yet known.

11. The process configuration for this bacterial fermentation process will require four main steps.

    a. pre-sterilization
    b. seed line
    c. process fermentation
    d. post-pasteurization or sterilization

12. Conceptual control techniques have been successfully developed and operated in the pilot facility.

    - nitrite monitor
    - pH monitor and control
    - integral P.C. controlled process

13. Major equipment requirements have been established. However, evaluation of the specific items has not yet begun.

14. Work on analytical requirements and procedures are underway. However, all of these are not yet available.

15. It is the opinion of our consultant (R. Hatch) that approximately 60% of the required data for process definition has been obtained. The remaining 40% consists primarily of fine details, process optimization and successful repitition of results and confirming data.

PLANS

1. Make necessary pilot plant modifications to be able to control contaminants.

2. Define the impact of variations in feedstock (nitrate and % solids) on the denitration process using ONCEL and diluting to desired concentrations.

3. Verify the reliability of the new startup procedure, using pH adjustment and inoculum grown and harvested in sterile SEL.

4. Define nutritional requirements for growth and growth inhibitors for the desirable organism. A future study will also be conducted to define the presence and variations of both nutrients and inhibitors in the SEL feedstock.

5. Identify and evaluate required process equipment.

6. Produce 3000 to 4000 pounds of RL sheet denitrified by the bacterial fermentation process to conduct product evaluations and mail out tests.

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7. Define the effluent gas composition and related environmental impact and control requirements.

8. Once acceptable results are achieved from the pilot plant, a sheet run will be made at Park 500 with SEL denitrated by bacterial fermentation. This is to better determine the product quality and acceptability.

9. A consultant firm with primary expertise in the developments of bacterial fermentation processes, will be identified and brought in to work with PR personnel on the development, scale up, and engineering of the N.O.D. process.

IV. DISCUSSION

Until late Fall 1981, the denitration pilot plant at A&O was using SEL from Park 500 and achieving good levels of denitration. The process was only operated in the feed-batch mode and batch times were in the range of 3 to 4 hours. However, the pilot facility did not provide much in the area of controls and flexibility. Also, the equipment configuration was not desirable, because it did not provide the ability to run the process like a fermentation plant should be run.

For these reasons it was decided that a new pilot facility was necessary. The new facility would provide more automation, controls and flexibility, and would be in a configuration more like a bacterial fermentation production facility. This would consist of four (4) steps:

A. pre-pasteurization
B. seed line
C. fermentation process
D. post-pasteurization

From November 16, 1981 through January 18, 1982 this new pilot facility was designed and built.

Once the equipment startup problems were resolved, several attempts were made to startup the seed line process with no success. The procedure used for this startup was the same that had been used for previous startups, but this time it would not work.

After approximately six unsuccessful attempts over a three week period, a difference in pH response was observed. It was noticed that each time the culture was transferred from the synthetic growth media to SEL, nitrate would start being converted to nitrite, pH would drop below six (6) and a failure resulted. Figure 1 is the pH curve during each unsuccessful startup attempt.

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When Figure 1 was compared with Figure 2, the pH curve for successful startup, instead of a steady drop in pH, a slight dip followed by a rise to a point of constant pH was observed. This was followed by the consumption of nitrite and successful denitration.

For the next startup the pH was adjusted so that it would not drop below 6.5. This resulted in a successfuly startup.

At this time, the exact cause of the problem is not known. What is known is that if the organism is grown and harvested in a media other than SEL they will adapt their metabolism for that media. When transferred to an SEL as media, these organisms will require some time to adapt their metabolism.

A new startup procedure is now being used. To reduce the time required to adapt the organism to a new media, the organism to be used for inoculum is grown and harvested in sterile SEL, concentrated and put into frozen storage for use when a startup is necessary. The new procedure also consists of initial pH adjustment of the startup media. This procedure has been successfully repeated several times in the lab but only once in the pilot plant. The previous best startup was 18 hours and the first time with the new procedure in the pilot plant it took 6 hours to startup.

Until the startup of the new pilot facility, almost all the work had been done with a mixed culture, consisting of many types of organisms. Since then, only pure cultures (single organism culture) have been used and significant efforts directed at maintaining the desired single organism predominant in the seed line.

Consistent dilution rates (rates of denitration) have not been achieved. This is primarily attributed to variations in SEL feedstock and contaminating organisms. Even though no data exists yet to substantiate that feedstock variations (nitrate and % solids) affect denitration, some indications are that it does. A test program to test this using DNCEL (4200 ppm NO_3N and 46% solid) and diluting it to the desirable concentrations is planned.

The redesigned pilot facility has added more sophistication in controls and data acquisition. Also the R&D analytical support on the project has been increased to provide more detailed up-to-date data (organic acids, nitrate, nitrite, pH, etc.)

With this added flexibility R&D has been able to determine that contaminating organisms exist in the process and these produce high levels of organic acids (acetic, butyric, and proprionic). The presence of the contaminants appears to be tolerable up to a certain level, but in excess can significantly upset and even wipe out the process.

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Significant amounts of data exist that the presence of contaminating organisms not only impact significantly on the rate of denitration but also produce extremely high levels of organic acids, primarily butyric acid, which introduces an extremely objectionable odor and taste to the product. Subjective testing is currently underway on sheet made with SEL denitrated in the pilot plant during the month of February. This testing will provide some data on what levels of these organic acids will be acceptable.

For this reason it is now unanimously accepted that sterilization of the entire SEL feedstock to the process will be required as well as means to maintain contaminants at or below critical level. The new pilot plant was not designed with the capacity to sterilize the entire SEL feedstream, only approximately 10%. This was because pasteurization of the rest of the feedstream was thought to be adequate. It was also felt that this process fermenter would not require provisions to keep airborne contaminants and others out because the desired organism would be kept predominant with a constant feed from the seed line. Therefore, the process fermentor was specified as an open vessel and all associated equipment designed with maintenance of sterility not considered.

From the data now available on contaminants, organic acids, sterilization and pasteurization will be necessary to sterilize the entire feedstream to minimize the introduction of contaminant organisms throughout the process. To do this, several modifications will be required to the pilot plant.

Approximately three months ago, R&D brought in a micro-biologist (Dr. Peter Kusel). Dr. Kusel has identified the need for the completion of a significant amount of work to better define the requirements of this particular bacterial fermentation process.

In order for the organism to grow, it needs nutrients which are for the most part in SEL. We know this because often the organism grows at tremendous rates in SEL. What is now required for better control of the process is to determine what the organism needs to grow and then determine the presence and variation in levels of these nutrients in the SEL feedstock. In conjunction with this, there are also growth inhibitors that need to be identified and their presence in SEL established. Plans to carry out this work are underway.

Many of the major equipment requirements have been identified and in several instances options exist. However, evaluation of specific equipment items has not yet begun.

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Currently two consultants with expertise in bacterial fermentation processes and technology are being used. Dr. Harry Wang, Professor of Chemical Engineering at the University of Michigan, and Dr. Randy Hatch, Associate Professor of Biochemical Engineering at the University of Maryland. Both consultants are very familiar with this process and the specific work done. After receiving the latest data, they are of the opinion that at least 60% of the work required to define a process has been completed. The remaining work consists primarily of working out the fine details, process optimization, equipment selection and evaluation and successfuly repetition of the results and confirming data.

Fermentation technology (scale up, design and engineering) is a new field for P.M. and as such, in house expertise is non existent. Plans are underway to get some engineering personnel trained on the basics of Fermentation Technology this Summer at M.I.T. However, this is only and introduction to basics.

Since PM Engineering's expertise is lacking significantly in fermentation technology, and since the R&D expertise in the same field is also limited, a consultant firm is being sought that can fill the gap.

The chosen consulting firm will work with both R&D and Engineering to insure the necessary data is generated in the pilot facility, properly analyzed and scaled up.

As of this writing, a significant effort has been put forth on this task and a list of 16 potential candidates has been narrowed down to four.

The current project priorities are:

A. <u>In the pilot plant</u>

1. Make required modifications to be able to control contaminant.
2. Evaluate impact of NO_3N and solids concentration on the process.
3. Successfully demonstrate ability to reliably startup.

B. <u>In the lab</u>

1. Define the nutrients and inhibitors for the growth of the organism.
2. Develop a reliable startup procedure.
3. Complete the study on the chemistry of SEL.
4. Identify effluent gas composition of this process.

The organization, scheduling and priority setting for this work is the subject of bi-weekly meetings of the entire project team. During this meeting the process definition and development items list is reviewed, updated and priorities set.

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United States Patent Office 3,420,241 Patented Jan. 7, 1969

3,420,241
METHOD OF PREPARING A RECONSTITUTED TOBACCO SHEET EMPLOYING A PECTIN ADHESIVE

John D. Hind and Robert B. Seligman, Richmond, Va., assignors to Philip Morris Incorporated, New York, N.Y., a corporation of Virginia

No Drawing. Application June 16, 1966, Ser. No. 557,903, which is a continuation-in-part of application Ser. No. 336,009, Jan. 6, 1964. Divided and this application Apr. 28, 1967, Ser. No. 647,277
U.S. Cl. 131—140 1 Claim
Int. Cl. A24b 1/14

ABSTRACT OF THE DISCLOSURE

This disclosure relates to a process for producing a binder composition for use in the manufacture of reconstituted tobacco. The binder is made from tobacco plant parts and involves the use of the naturally occurring tobacco pectins, which are obtained by a process in which ammonium orthophosphate is employed to treat the tobacco plant parts. The treatment involves the destruction of the alkaline earth metal cross-links of the tobacco pectins, the release of the resulting tobacco pectins by a washing action and the depositing of the released tobacco pectins on the treated plant parts.

This application is a division of application Ser. No. 557,903, which was filed on June 16, 1966 now Patent No. 3,251,361, and which, in turn, is a continuation-in-part of application Ser. No. 336,009, which was filed on Jan. 6, 1964, now abandoned and which, in turn, is a continuation-in-part of application, Ser. No. 240,130 filed Nov. 26, 1962, now forfeited and application, Ser. No. 169,895 filed Jan. 26, 1962, now abandoned.

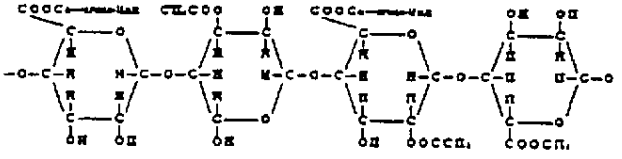
This invention relates generally to a method for the production of an adhesive tobacco composition in which tobacco pectins within the tobacco itself serve as the binder.

During the production and processing of tobacco products, including aging, blending, sheet forming, cutting, drying, cooling, sweetening, shaping and packaging, considerable amounts of tobacco fines and tobacco dusts are produced. It is known that such tobacco fines and dust can be combined with a binder to form a coherent sheet, which resembles leaf tobacco and which is commonly referred to as reconstituted tobacco. One method for making reconstituted tobacco of this general character is disclosed in United States Patent No. 2,734,510, wherein the tobacco fines and dust are applied to a binder made of carboxymethyl cellulose carboxymethyl hydroxethyl cellulose or a suitable salt thereof. The binder, in such compositions, ranges from about 5% to about 50% of the weight of the tobacco employed. United States Patent No. 2,706,175, describes a binder for reconstituted tobacco which consists of a plant gum, principally of galactomannan. United States Patent 2,592,554 to Frankenburg describes, as binders for reconstituted tobacco, various water-soluble polysaccharides, such as alginic and pectinic acids and their sodium and potassium salts, derived from plants other than tobacco, for example derived from citrus fruits. However, the addition of cellulosic binders further increases the amount of cellulosic material in the product and tends to create an acrid and bitter smoke, when the product is used to make cigarettes. The natural hydrophilic colloid gums such as guar gum, locust bean gum, algin and other commonly used materials, such as Irish moss, have additional disadvantages. These materials contain proteins and other materials not found in tobacco which add distinctive flavors of their own to tobacco products during smoking. Thus, Frankenburg, in describing the use of various water-soluble polysaccharides derived from plants other than tobacco, teaches that care should be exercised that they must be in a state of refinement. Frankenburg teaches that these materials should be free of extraneous matter containing compounds of nitrogen, particularly proteins, and compounds of sulfur, phosphorus and the halogens; i.e., compounds giving undesirable products of combustion or dry distillation. Such refining is often a very tedious and difficult operation.

The present invention makes possible the production of improved reconstituted tobacco by a method which is simpler and more effective than the methods previously employed. The present method does not require refining of the binder and it, therefore, more easily and efficiently employed than other methods for making binders and for making reconstituted tobacco. The reconstituted tobacco which is obtained in accordance with the present invention need not contain any additional cellulose or proteinaceous material which is foreign to tobacco, since the binder which is employed may be derived solely from tobacco, and may be produced in such a manner that it contains no materials other than those which naturally occur in tobacco. Thus, reconstituted tobacco produced in accordance with the invention, can be so formulated as to be similar in physical properties and chemical composition to natural tobacco.

The term pectic substances[1] will mean those substances which are found in many plant products, and which consist essentially of partially methylated galacturonic acids joined in long chains.

The pectic substances found in tobacco plants contain acetyl groups and differ considerably from commercially available pectins found in other plants, including sugar beet pectins and citrus and fruit pectins. Tobacco protopectins are uniquely insoluble in hot water as compared with protopectins from many other sources and comprise mainly water-insoluble pectins (protopectins) consisting of the calcium and magnesium salts of partially esterified and slightly acetylated polymers of galacturonic acid. The divalent calcium and/or magnesium atoms act as cross-links between acid chains, thus making the polymers water-insoluble. As an illustration, the structure of the calcium salt of a polymer of galacturonic acid can be represented as above.



[1] Note: Unless otherwise specified, the term "pectins" will, for convenience, hereinafter be employed interchangeably with the term "pectic substances."

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Although pectins have long been known as constituents of plant tissue, it has been found extremely difficult to separate pectins from the remainder of plant compositions and to obtain them as homogeneous compositions. The recovery of pectins from tobacco is even more difficult than the recovery of pectins from other plants.

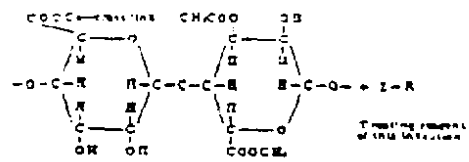
In accordance with the present invention, tobacco parts are bonded together by tobacco pectins which are especially prepared by a novel process which yields these pectins in a form in which they can be employed as binder materials. Our process for preparing tobacco pectins comprises first reacting tobacco parts, preferably in a form in which they present a large surface area, with an aqueous solution of a non-ionic reagent which is capable of reacting with and destroying the calcium and magnesium cross-links in the pectinaceous substances which naturally occur in tobacco. After the calcium and magnesium cross-links are destroyed, the tobacco pectins are liberated and made available for use as a binder. The tobacco pectins are then dissolved or dispersed in solution, or are at least sufficiently released from the interstices of the tobacco mass so that they form a coating on the surface thereof. Tobacco pectins which are dissolved or dispersed in the treating solutions are thereafter precipitated or deposited from the solution, so that they become available for use as a binder material. In this way, the tobacco parts can be bonded together by a binder material which is made of ingredients that are closely related to the naturally occurring ingredients of tobacco. The bonding can be accomplished without the need for the purification of the tobacco pectins, inasmuch as any materials which are present are closely related to the materials which are normally present in tobacco and, thus, do not add any undesired qualities to the tobacco.

The tobacco parts which can be employed in the present invention including tobacco leaves, stems and stalks, or a mixture of these, whether in sheet, flake, particulate or other form. Preferably, the parts are ground, cut or otherwise prepared in a form which presents a large surface area. The portions of the plant comprising the stems or midribs, and often referred to as tobacco petioles, are the preferred starting materials. Tobacco stalks contain lesser amounts of pectinaceous materials but can also be employed.

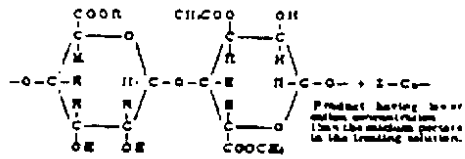
In the first step of the process of our invention, tobacco pectins are liberated from pectinaceous materials in tobacco by reacting the pectinaceous materials with a reagent which, under the conditions of the reaction, is reactive with the calcium (and/or magnesium) contained in them to form a compound or product having a lower calcium ion, and, in the case of magnesium, magnesium ion, concentration[1] in the treating solution than the naturally occurring calcium (or magnesium) pectate. This reagent may, for convenience, be hereinafter referred to as a "cross-link destroying reagent."

The reaction may be generally represented by Equation I, which illustrates the reaction of one type of tobacco protopectin (a calcium salt of a polymer of galacturonic acid), wherein calcium cross-links are present with the reagent of the present invention. In the equation, R may be hydrogen, in which case the product is pectic acid, or R may be a monovalent cation, such as sodium, potassium or ammonium, in which case the product is a soluble pectate.

[1] By concentration we mean concentration or activity as set forth in Glasstone, "Textbook of Physical Chemistry," 2nd Edition, p. 934, D. V. Nostrand Company, Inc.



CALCIUM PECTATE



PECTIC ACID OR SOLUBLE PECTATE

In one embodiment of this invention, the reagent, which can be, and preferably is, in aqueous solution, acts by forming a precipitate with the calcium or magnesium, in which case, it can be a water-soluble monovalent metal salt of the formula M_nX wherein M is a monovalent inorganic cation, n is an integer having a value of 1, 2, or 3 and X is an anion which may be monovalent or polyvalent, such that the calcium salt of the formula Ca_pX_q is essentially insoluble in the treating solution and p and q are integers corresponding to the functionality of X. Monovalent cations which are effective include the alkali metals such as sodium, potassium and lithium, and also include such monovalent cations as ammonium, and substituted ammonium ions (NR_4^+, where R=aryl or alkyl). The anion portion of the molecule may be $CO_3^{=}$, $PO_4^{\equiv}$, $HPO_4^{=}$, $H_2PO_4^{-}$, and the like. For example, the compound M_nX could be sodium carbonate, Na_2CO_3, since sodium is a monovalent inorganic cation and calcium carbonate is essentially water insoluble. Additional representative examples of precipitating agents are the orthophosphates, metaphosphates and carbonates of sodium, potassium, lithium and ammonium. In the case of the orthophosphates, the anion portion of the molecule may be either $PO_4^{\equiv}$, $HPO_4^{=}$, or $H_2PO_4^{-}$. Specifically, for example, when ammonium orthophosphate is used, the precipitate is calcium and/or magnesium ammonium phosphate. The pH of this reaction should be between about 5.8 and 10 and the temperature may be as high as 400° C. but should, preferably, be between about 25° C. and about 135° C. for a period of from about 1 minute to about 24 hours. Preferred precipitating agents which may be employed are the alkali metal carbonates, for example, sodium carbonate and potassium carbonate. Particularly preferred precipitating agents which may be employed are the alkali metal phosphates and, most particularly, the alkali metal orthophosphates and the ammonium orthophosphates, such as the ammonium phosphates and ammonium orthophosphate, sodium orthophosphate, potassium orthophosphate, sodium dihydrogen orthophosphate, ammonium dihydrogen orthophosphate, potassium dihydrogen orthophosphate, diammonium monohydrogen orthophosphate, disodium monohydrogen orthophosphate and dipotassium monohydrogen orthophosphate.

In a second embodiment, the cross-link destroying reagent acts by sequestering the calcium or magnesium, thereby removing the calcium or magnesium atoms by forming a complex therewith. Suitable reagents of this type include any sequestering agent which will form a complex or chelate with the calcium and/or magnesium, thereby removing the calcium and/or magnesium and making them unavailable for cross-linking with the pectin. Illustrative of such sequestering agents are ethylene-diamine-tetraacetic acid and similar amino acids, alkali

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metal poly-metaphosphates such as tetra-metaphosphates, hexametaphosphates and trimetaphosphates, pyrophosphates and tripolyphosphates, such as sodium hexametaphosphate, tetrasodium pyrophosphate and pentasodium tripolyphosphate. The mechanism which occurs when a sequestering agent is employed is the formation of a chelate; calcium and magnesium ions are no longer available to combine with the pectate ions in solution. Many naturally occurring amines and peptides are also effective as sequestering agents for calcium and/or magnesium. Representative examples include alanine, aspartic acid, glycine, glycyl glycine, glutamic acid, serine, l-tyrosine and di-iodo-l-tyrosine. Amino acids that are effective as chelating solubilizing agents include beta alanine, N,N-diacetic acid; amino barbituric acid, N,N-diacetic acid; 2-amino-benzoic acid, N,N-diacetic acid; beta-aminoethyl-phosphonic acid, N,N-diacetic acid; beta-aminoethylsulfinic acid, N,N-diacetic acid and ethylenediamine-tetra-acetic acid. The pH of this reaction should, preferably be between about 7 and about 10 and the temperature should, preferably be between about 0° C. and about 145° C. for a period of from about 1 minute to about 24 hours.

A cross-link destroying reagent may also function partially as a pectin-removing reagent, in accordance with the first embodiment of the invention, and partially as a sequestering agent, in accordance with the second embodiment. Such a reagent, for example, is diammonium monohydrogen orthophosphate (DAP), which is a particularly preferred material, in accordance with the invention.

In a third embodiment, the cross-link destroying reagent is an acid wash which forms the released but insoluble free pectic acid and soluble calcium and magnesium salts. Generally, the acid wash will comprise an inorganic acid, such as hydrochloric acid, phosphoric acid, sulfuric acid or a similar acid, which will form soluble calcium and magnesium salts under the following conditions. Hydrochloric acid and sulfuric acid are particularly preferred. The acid may be employed as 0.25 N to 5.0 N solutions, but is preferably employed as 0.5 N to 1.0 N solutions. The exact solution and amount to be employed will vary with the particular acid which is used, it only being necessary that sufficient acid be present to convert the calcium and magnesium present in the tobacco being treated to the calcium and magnesium salts of the acid. The acid treatment is preferably conducted at a temperature of from about −1° C. to about 30° C. The acid treatment comprises reacting the tobacco parts with the acid until the resulting mixture has a pH of from about 2 to about 3.5. Preferably, the pH is brought to from about 2.0 to about 3.0, the most desirable pH being between 1.... and 1.55. This treatment will generally be conducted from about 10 minutes to 24 hours, depending in part on the ... of the tobacco particles. The acid conditions which are necessary for this embodiment of the invention may be achieved by ... of ion exchange resins which may be used, with suitable recycles, to obtain the desired pH of the solution during treatment. The ion exchange resins may be used and regenerated in accordance with the usual practices for such resins.

Preferably, the mixture resulting from the acid treatment is then washed with water. This water wash step is preferably conducted at a temperature of from about 15° C. to about 35° C. and, preferably, distilled water is employed. When this wash step is employed, sufficient water should be used to remove the calcium and magnesium salts of the acids, which salts are formed in the above-described treatment, thus, there should be at least 2 volumes of water per volume of the mixture resulting from the acid treatment. The wash water is separated from the tobacco by any suitable means, for example, by conducting the wash in a centrifuge, filter press, Buchner funnel, or any other apparatus from which liquids can be substantially removed from solid materials.

In summary, in the first step of the first embodiment of this invention the treating agent Z–R attacks the calcium and/or magnesium cross-links of tobacco protopectin and forms a precipitate which is a salt of calcium and/or magnesium, thus removing the calcium and/or magnesium from the protopectin and from the solution. In the first step of the second embodiment, the treating agent Z–R is a sequestering agent which forms a chelate of the magnesium and/or calcium from the tobacco protopectin and makes the calcium and/or magnesium unavailable for recombining with the pectins. With certain reagents, such as DAP, the first step of the present process may comprise a combination of the mechanisms of the first embodiment of this invention and the mechanism of the second embodiment of this invention. In the first step of the third embodiment of this invention, the treating agent Z–R is an acid which attacks the calcium and/or magnesium cross-links of the tobacco protopectin and forms the soluble calcium and/or magnesium salts, which are then washed away from contact with the pectins.

In the first step of each of the first two embodiments of the invention, the pectin which results is in condition for release from the tobacco cell structure, R in Equation I being a monovalent inorganic cation such as sodium. In the first step of the third embodiment, the insoluble pectic acid resulting from the acid treatment must be reacted with an alkaline material before it is in condition for release from the tobacco cell structure.

The acid treated pectins are placed in condition for release by bringing the mixture resulting from the acid treatment, and preferably, after the water wash described above, to a pH of from about 5.0 to about 10.5 and, preferably, from about 6.3 to about 8.5, by the addition of an alkaline material. Suitable alkaline materials include ammonium hydroxide and alkali metal hydroxides, for example, sodium hydroxide, potassium hydroxide and lithium hydroxide, and alkali metal salts, such as sodium bicarbonate, sodium carbonate, sodium phosphate, and similar salts to convert the pectic acid to a soluble form. The alkaline material may be any water-soluble compound containing a monovalent inorganic cation and capable of producing hydroxide ions when dissolved in water. The temperature of this step may be from about −1° C. to about 45° C., but is, preferably, from about 15 to about 35° C. The alkaline material is preferably in the form of solid particles or in the form of a solution having a concentration of from about 5 to about 50%.

Once the tobacco pectins have been liberated from the tobacco, by the removal of the calcium and magnesium cross-links, they should be released from the interstices of the tobacco. That is, they will be made available to the solution or suspension or, in certain instances, they will be merely deposited on the surface of the tobacco particles. This comprises the second step of the process of the present invention. In embodiments 1 and 2 of the invention, this release or second step may be accomplished concurrently with the first step by reacting with the solution of the treating reagent. In embodiment 3, however, as indicated above, the insoluble pectic acid resulting from the treatment should be reacted with an alkaline material before it can be released. In such event, the release may be concurrent with the addition of the alkaline material due to a washing action. In any case, additional treating liquid or water may be used to effect the release through a washing action of the treated tobacco particles.

In accordance with the third step of our process, the liberated and separated tobacco pectins can next be precipitated or deposited in a relatively free form (as compared with the tobacco pectins as they were originally present in the tobacco), from the treating solution, for example, by being formed into the insoluble pectic acid or into an insoluble salt of pectic acid or by the action of a water-miscible solvent, such as acetone or ethyl alcohol to cause a water-soluble salt of pectic acid to go out of so-

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lation. In the case of tobacco pectin solutions such as solutions of sodium and potassium pectinates and/or pectates that can be accomplished by acidifying the solution until the pectins precipitate or by adding a gelation agent, such as an alcoholic solution, preferably having a pH of from about 1 to 9 and, preferably, from about 1 to 5. The pH of the alcoholic solution can be regulated by the addition of a mineral acid, such as HCl, to the alcohol. Although the preferred gelation agent is ethanol, any water-miscible organic solvent having up to about 10 carbon atoms may be employed, for example, a ketone, such as acetone, or a diether, such as dioxane. Water-immiscible solvents such as ether, for example, ethyl ether, can be used, if combined with a water-miscible solvent, such as acetone.

The tobacco pectins can be recovered by concentrating the solution or suspension in which they are present until they precipitate. This precipitate might also be characterized as an intractable mass, since the pectin solution, upon concentration, generally becomes progressively more viscous until it finally dries to leave a deposit in a glassy solid state.

While the tobacco pectins can be separated and purified before use as a binder in reconstituted tobacco sheets, they are preferably employed just as they are produced in situ, i.e., in combination with the treated tobacco plant parts from which they were obtained, the entire combination comprising the binder for reconstituted tobacco sheets or, under some circumstances, the entire combination comprising essentially the entire components of a reconstituted tobacco sheet. By using the entire mixture, no original tobacco flavors are lost, all of the tobacco is employed, and no expensive and time-consuming refining operations are required.

Although it is not necessary, the thixotropic properties of solutions containing soluble pectins can be adjusted in the preparation of a cured sheet by the addition of such materials as calcium chloride. If any complex or precipitate formed in the first step of this process is present with the soluble pectates, the thixotropic properties of the mixture can also be adjusted by adjusting the pH to precipitate calcium and magnesium pectates.

A preferred preliminary step, in accordance with the present invention, comprises washing the tobacco plant parts which are preferably ground or cut to a relatively small size, with cold water. This water wash serves to remove impurities which might otherwise hinder the subsequent treatments in accordance with the present invention. It is particularly desirable to employ such a cold water washing step when alkali metal carbonates are employed as the reagent in the first step. Generally, sufficient water should be used during such a water wash operation to cover all of the tobacco plant parts present. The temperature of the water may be between −1° C. and 100° C., but is preferably about 20° C., and the water wash should generally continue for a period of from about ½ to 2½ hours. Agitation during the wash is desirable, but not necessary. After the water wash has been completed, the water can be removed from the tobacco parts by filtration, decantation or other suitable means.

As discussed above, a particularly preferred embodiment of the present invention involves the use of an ammonium or alkali metal orthophosphate, such as diammonium monohydrogen orthophosphate (DAP), for the release of the tobacco pectins. The DAP will, generally, be added to the tobacco plant parts, which may, for example, be bright tobacco parts, burley tobacco parts, or a mixture of the same, in an aqueous solution. The concentration of the DAP in the aqueous solution is not critical, but will, generally, be in the range of 0.5–5.0% by weight. The DAP and water may be added separately to the tobacco. The amount of DAP should, preferably, comprise from about 0.01 to about 0.5 part, and, most preferably, from about 0.03 to 0.35 part (by weight) per part of tobacco being consulted. A humectant, such as glycerin or triethylene glycol, may be present, if desired, at about 0.5 to 1 per part of DAP. The temperature during the DAP treatment of the tobacco may vary between room temperature and about 190° F. or higher, depending on the type of tobacco being treated. Under pressure, even higher temperatures may be employed. The pH of this mixture is, preferably, maintained at a value about 7.1 to 9.0, which may conveniently be accomplished by the addition to the solution of concentrated aqueous ammonia. By agitating or stirring such a mixture under the above-described conditions for from about 1 minute to about 1 day, and, preferably, from about 1 hour to about 5 hours, the tobacco pectins are liberated, released and deposited in the tobacco plant parts to form a binder composition. The resulting mixture may then be refined, for example, in a disk refiner, until substantially all the pulp (in excess of the water present), can be shaken through a screen of approximately 18 mesh to produce a binder composition which is ready for use in the manufacture of reconstituted tobacco sheets.

The term "tobacco pectins" as used throughout this specification means "liberated tobacco pectins" and comprehends pectins which have been freed or liberated from tobacco and are, therefore, not bound into the tobacco structure, as differentiated from the insoluble, naturally-occurring protopectins which are found into a plant cell structure. The term includes the free pectinic or pectic acid, as well as soluble salts such as the sodium, potassium, ammonium, pectates and pectinates, and insoluble salts such as the calcium and magnesium pectates and pectinates depending on what method is employed to liberate and obtain them from the naturally occurring insoluble protopectins.

The tobacco pectins produced or liberated in situ or isolated by means of this invention can be used as the sole binder material for reconstituted tobacco, i.e., no other materials need be added to make the sheet. They can be sprayed, extruded or cast, thus facilitating applications onto a moving belt carrying tobacco dust. Under proper conditions of formulation and processing, reconstituted tobacco made with the tobacco pectins produced by this invention exhibit excellent physical and aromatic properties. The ultimate tensile and wet strengths of the reconstituted tobacco are good. While no other materials need be added to the pectinaceous binder, other materials can be added, if desired. For example, organic acids and preservatives which may in themselves be of tobacco origin, may be added. Plasticizers, such as glycols and polyglycols, and humectants, such as glycerin, may also be added, if desired. In addition, the gel strength of the tobacco pectins can be regulated by partial precipitation to control such rheological properties as viscosity, fluidity and elasticity. Other additives or dispersants may be added in small amounts to regulate slurrying qualities, provided, however, that such substances are not added in large enough quantities to adversely affect the flavor or aroma of the final product. Furthermore, the tobacco pectins can be combined with water-soluble gums or water-dispersible gums commonly used as binders for tobacco sheets such as methyl cellulose, sodium carboxymethyl cellulose, guar gum, locust bean gum, or alginates, although it is preferred to minimize or eliminate such additions in order to obtain a product which most closely resembles natural tobacco.

The product from treating the tobacco plant parts in accordance with the methods of the present invention may be cast directly and dried and cut into particulate material similar in physical form to ordinary smoking tobacco and so used, preferably mixed with tobacco leaf cut or shredded in the usual manner. The product may be cast in sheet form, in blocks or as threads or other shapes, as desired. An important use, however, of the prepared composite slurry or easily molded isolated pectinaceous mass is as a binder for ground tobacco and for the making of corresponding tobacco products suitable

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for smoking. Sheet material of widely different properties may be formed by suitable variations in the manner of forming. One method and product comprises flowing the composite slurry onto a moving belt and applying a layer of dry ground or fragmented tobacco to the wet adhesive surface. If desired, there may be first applied to the belt a layer of the tobacco, followed by a layer of the binder, and then a top layer of the tobacco. Various additives may be included with the ground tobacco such as flavorants, plasticizers and aromatic substances. The web is ultimately dried and then suitably moistened and rolled up. Such methods of forming continuous sheets are known generally in the art and the details need not be further described. Representative of this procedure is the apparatus and method disclosed in U.S. Patent 2,734,511.

Another method of forming a reconstituted tobacco product, with the slurry of the isolated tobacco pectins as a binder, comprises mixing ground tobacco thoroughly therewith into a mass of dough-like consistency and then casting the mass in sheet form onto a moving belt surface followed by drying and remoistening in accordance with the known procedures. Representative of this procedure is the apparatus and method disclosed in U.S. Patents 2,703,175 and 2,769,934. Obviously, the reconstituted tobacco may also be formed by molding or other suitable means.

A particularly preferred aspect of the present invention comprises employing, as a binder or directly, the mixture of tobacco and tobacco pectins which have been produced in situ, without any separation steps and without the necessity for any additional adhesive materials.

The following examples are illustrative:

Example 1

Tobacco stems (100 parts) were covered with cold water and leached for one hour. The water was then decanted and discarded. A leaching solution, made from 1 part of sodium carbonate dissolved in 40 parts of water, was added to the leached parts. This mixture was heated at its boiling point for 30 minutes at atmosphere pressure and then for 20 minutes at 20 p.s.i.g. In the course of this treatment the tobacco pectins were dissolved from the tobacco parts. The entire wet mass (pulp mixture) was dried and ground in a Waring Blendor so that it would pass through a 20 mesh screen. The resultant material had gelling properties and was thixotropic in nature.

To this material was added 2 parts of glycerin, to serve as a humectant. The pH of the resulting mixture was adjusted to 6.0 by the addition of a solution of 10% hydrochloric acid. One gram of sodium carboxymethyl cellulose (CMC) was added to the mixture, giving a proportion of about 1 part of CMC to 10 parts of total solids in the mixture. Since it was desired to use this mixture as a sprayed tobacco binder, the CMC was employed in order to adjust the spraying qualities. The mixture of (a) treated tobacco stem parts, including the liberated tobacco pectins, (b) the sodium carboxymethyl cellulose and (c) the glycerin had a viscosity which was suitable for spray application of the mixture as a binding material for reconstituted tobacco. This material was sprayed on top of an undercoating of tobacco dust, of an 80 mesh size, which had been dusted on a wet belt. Another coat of the tobacco dust was laid on top of the binder. In general an apparatus similar to that described in U.S. Patent 2,734,513 was employed. As in said patent, the reconstituted sheet was dried and was then humidified to the desired moisture content. The control in Table I was made in a conventional manner, using a relatively large amount of CMC in proportion to the pulped washed tobacco stems. The test data as observed are recorded in Table I.

TABLE I

Sheet No.	[illegible]	[illegible]	[illegible]	[illegible]
Part CMC [1]	1:10	1:10	1:20	1:20
Moisture (percent) [2]	12.3	12.1	12.1	12.1
Tensile, Gr./in. [3]	630	710	490	520
Elmendorf Tearing Test [4]	71	74	81	79
Elongation (percent) [5]	3.3	3.3	3.8	4.0
Burning Rate, sec./cm. [6]	4.8	4.9	5.1	5.7

[1] Part CMC per parts of total tobacco solids in the slurry (including tobacco portions).
[2] The percentage of moisture contained in the entire sheet (wet basis).
[3] The tensile strength of a 10 mm. test strip when it is under an average load in grams.
[4] One test load strip obtained by application of a 100 gram weight for 30 seconds.
[5] The percent elongation at breakage on the Instron test machine.
[6] Free burning rate of a sample test strip, in air.

The reconstituted tobacco sheets made pursuant to this example were shredded and made into cigarettes. The test cigarettes, as well as control cigarettes made from standard reconstituted tobacco sheet, were submitted to a smoking panel for subjective testing. The panel found that the smoke of the cigarettes made by the method of this invention was significantly less harsh than the smoke obtained from the control cigarettes. A pleasing vanillin odor was found to be transmitted into the aerosol phase of the smoke from the test cigarettes made employing reconstituted tobacco made by the present invention.

Example 2

Fifty grams by weight of burley tobacco stems were thoroughly washed in about 5 liters of cold water for three hours. The washed tobacco stems were then mixed with 500 grams of water having dissolved therein 5 grams of sodium carbonate. The resultant mixture, containing about 8% solids, was steamed under atmospheric pressure for 30 minutes and under a pressure of 20 p.s.i.g. for an additional 20 minutes. At the end of this time, the mixture was allowed to cool and the liquid was separated from the solid materials. The solid materials were treated in a cider press to recover as much as possible of the remaining liquid. The liquid was added to a previously prepared coagulant bath which consisted of ethanol and hydrochloric acid, in an amount to adjust the pH of the coagulant to about 1.0. The resulting mixture (which had a pH of about 3.0) was alternately stirred and allowed to settle for a period of two hours. At the end of this time the mixture was strained through a cloth sieve and the filtrate was discarded leaving a solid, gel-like mass, consisting essentially of pectinaceous materials combined with approximately 10 parts of liquor. The yield of pectinaceous materials was 15%, based upon the dry weight of the starting tobacco plant parts. The mass was observed to be thixotropic, soluble in water at a pH of about 6 and soluble in a sodium carbonate solution.

The tobacco pectins thus isolated were quite impure and had a color characteristic of tobacco. The solid mass was then redissolved in a sodium carbonate solution and the resultant solution was poured into an acidified ethanol bath similar to the coagulant bath used earlier. The recoagulated solid was obtained by filtration and dried in an oven at 105° C. The dried tobacco pectins were in the form of an opaque, substantially colorless sheet. When the sheet was pulverized, a white powder was obtained having a distinct and pleasing odor similar to vanillin.

Ten grams of the dried tobacco pectins prepared above were swelled in 100 ml. of cold water. The mixture was then added to a slurry consisting of 8.5 grams of refined kraft pulp dispersed in 200 ml. of water. The resultant mixture was homogenized in a Waring blendor. Four grams of glycerin were then added to the mixture to serve as a humectant. The resulting mixture was cast into a binder film, using a Gardner casting knife set to produce a sheet having a wet thickness of 10 mils. The physical properties of this sheet, after drying, were tested and were found comparable to the properties of conventional binder films. The physical test data are given in Table II. The control material was made by mixing pulp, which had

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been refined with NaOH, with sodium carboxymethyl cellulose (CMC) and a humectant, as is shown in Table II.

TABLE II

	Test binder-film	Control binder-film
Binder material	(1)	(2)
Basis weight, grams per sq. yd.	3.5	3.51
Moisture content	14.4	14.0
Tensile strength	0.58	0.67
Tensile energy absorption	3.0	3.3
Elongation, percent	6.1	6.1
Wet mullen, grams per sq. in.	27.90	32.0

[1] Dried tobacco pectins extracted from tobacco petioles.
[2] CMC (sodium carboxymethyl cellulose) and pulp as prepared by known methods and containing about 25% CMC.
[3] Wet mullen is proportional to the product of the tensile strength and the extensibility.

Example 3

One hundred grams of burley tobacco stems were covered with distilled water, leached for 30 minutes and then drained. The washed stems were then mixed with a solution containing 10 grams of diammonium monohydrogen orthophosphate in 600 ml. of water. The resultant slurry was heated for 1 hour at a temperature of from 90 to 100° C. The pH of the reaction mixture, after completion of the reaction, was about 7. The entire reaction mixture was homogenized in a Waring blendor. The solids content was determined to be 5% by weight.

Two grams of glycerine, to serve as a humectant, were blended with 200 grams of the above reaction mixture. A film of 50 mil wet-thickness was cast from this mixture. The extruded film was found to have the property of being able to slide off a surface even when newly cast. After the film was partially dried, it was found that it could be peeled off a plate by hand, while still partially wet and then hung up to dry like wet cloth. This property provides definite advantages in the manufacture of tobacco products.

Physical tests were carried out on the dried film thus prepared and were also carried out on a control film made by using enzymatically pulped tobacco plant parts with sodium carboxymethyl cellulose (CMC) as the binder. A film of binder as made in this example was burned and was found to give a pleasant aromatic smoke. The test data are given in Table III.

TABLE III

	Test binder-film	Control binder-film
Binder	(1)	(2)
Basis weight, grams per sq. yd.	3.5.9	3.6
Moisture, percent	14.3	14.0
Tensile strength	0.53	0.57
Tensile energy absorption	2.3	2.5
Elongation, percent	4.1	4.1
Wet mullen, gm./sq. in.	35.8	40.9

[1] Based on total solubility (excluding pectin gel formed in situ).
[2] CMC alone (pulp).
[3] Kg.-cm. per sq. cm. based on dry film.
[4] Kg.-cm. per sq. cm. wet film.
[5] Wet mullen is proportional to the product of the tensile strength and the elongation.

Examples 4–10

In each of these examples, burley tobacco stems were covered with distilled water, leached for 30 minutes, and drained. One hundred grams of stems were treated with the agent indicated in Table IV. The agent was dissolved in 600 ml. of distilled water in an amount sufficient to make up a 10% (by weight) solution. The leached tobacco stems were placed in said solution and heated in a steam bath for 1 hour at a temperature of from 90 to 100° C. (Except for the example in which Versene was employed. That example was conducted at room temperature.) A five ml. aliquot was taken from the resulting mixture and mixed with 20 mls. of ethyl alcohol in a graduated cylinder whereby a precipitate of tobacco pectins was obtained. The amount of precipitate in the graduated cylinder was observed. Each of these precipitates of tobacco pectins was suitable for use in the preparation of a reconstituted tobacco sheet in accordance with the methods taught by the present invention. The results obtained in these experiments are presented in Table IV below.

TABLE IV

Reagent:	Volume of coagulated tobacco pectins
Pentasodium tripolyphosphate	15
Sodium hexametaphosphate	5
Diammonium monohydrogen orthophosphate (DAP)	15
Trisodium orthophosphate	15
Disodium monohydrogen orthophosphate	15
Dihydrogen monosodium orthophosphate	5
Versene (tetrasodium salt of ethylenediamine-tetraacetic acid)	15

Example 11

One hundred grams of burley tobacco stems were covered with distilled water, leached for 30 minutes and drained. A solution of 10 grams of Versene (tetrasodium salt of ethylenediamine-tetraacetic acid) in 600 ml. of water was added to the stems and the mixture was made basic with aqueous sodium hydroxide. The resulting mixture was then heated for 1 hour at room temperature. The entire mixture (a pulp) was homogenized in a Waring Blendor and the solids content was determined to be 5% by weight. A 200 gram portion of the pulp, thus prepared, was combined with 2 grams of glycerine, as a humectant, and then cast into a film of 30 mils thickness by the use of a Gardner casting knife. The resultant sheet was found to have satisfactory physical properties and, upon burning, exhibited a very pleasing aroma.

Example 12

Green bright tobacco leaves were soaked in isopropanol to remove sugars and chlorophyll. The midrib was stripped out of the leaves leaving the web. One thousand grams of the web (82 grams on a solids basis) were placed in a porcelain bucket and covered with boiling distilled water containing 9.3 grams of diammonium monohydrogen orthophosphate (DAP). The DAP was added on the basis of 10 grams of DAP per 100 grams of stems (which contained 12% moisture, by weight). The resulting mixture was boiled at low heat for one hour. The juice was then first expressed from the mixture by hand. Solids, for example cellulose and sand, were then further separated from the juice by centrifuging. The clear juice from both operations was then mixed with 70% ethanol to form a gel, which was then squeezed out in cheese cloth. The gel was transferred to a Buchner funnel where it was washed first with acetone and then with ethyl ether. Finally, the gel was placed in a vacuum desiccator and dried. The gel was suitable for use in the preparation of a reconstituted tobacco in accordance with the teachings of the present invention.

Example 13

Burley tobacco stalks were separated into cortical tissue, woody tissue, and pith. A sample of each was steam cooked with 10% sodium carbonate for 30 minutes at atmospheric pressure and for 20 minutes at 20 p.s.i.g. After this cooking, the woody tissue was still hard and could not be pulped in a Waring Blendor. The cortical tissue and pith were soft and pulpable. Each of these latter two preparations were suitable for use in the preparation of reconstituted tobacco.

Example 14

Coarse ground bright stem tobacco fines (30 grams) were washed thoroughly in cold water, and then placed in a boiling aqueous solution of 3 grams of diammonium monohydrogen orthophosphate and cooked for 5 minutes. The mixture was then placed in a Waring Blendor.

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After a very short period in the blender, the mixture was converted to a viscous, fine, impalpable slurry, wherein the tobacco stem particles had been completely separated to units of cellular size. This impalpable mass was suitable for use as a binder in reconstituted tobacco. When reslurried with some of the cold water washings removed in the first step, the impalpable mass immediately became viscous and eventually jelled to a soft mass.

For further comparison, a similar sample of coarse ground bright stem fines (30 grams), was dispersed in boiling water and cooked for about 30 minutes with three grams of diammonium monohydrogen orthophosphate. The pH of this mixture was brought to a value of 7.1 by the addition of 30% aqueous ammonia. The granules of tobacco could then be refined to a palpable pulp, similar to the soft mass produced in the experiment described in the first part of this example.

Example 15

Burley tobacco stems (½ pound) were covered with distilled water, allowed to stand several hours and the water decanted. This step was repeated several times and finally the stems were covered with distilled water containing 30 ml. of concentrated HCl and left overnight at 24° C.

After standing overnight the acidic water was decanted and the stems washed repeatedly free of HCl until the wash water gave no precipitate when treated with silver nitrate solution. The stems were then covered with distilled water containing 25 grams of sodium carbonate and left overnight at 24° C. The pH of the sample, the following morning, was 8.8. The stems were swollen and soft and were easily disintegrated with the fingers and the mixture could be homogenized and employed, as a binder in reconstituted tobacco.

Example 16

The apparatus employed in this experiment was large scale equipment, consisting of a 200 gallon conical bottom, open top, stainless steel tank, fitted with a Cowles high shear mixer.

One hundred and forty-eight gallons of water were placed in the tank and heated to a temperature of 207° F. One hundred and twenty-eight pounds of bright tobacco (milled to pass a 6 mesh per inch sieve) stems were added, while operating the Cowles mixer at a low speed. Almost immediately after the addition of the tobacco, nine and one-quarter pounds of diammonium monohydrogen orthophosphate (technical grade) were added to the mixture. Ammonia (assaying 28%, by weight NH_3) was added to adjust the pH to 7.1. The mixing speed was increased to 2700 r.p.m. After a period of 3 minutes the temperature of the mixture was 194° F. Some of the particles in the mixture were soft enough to be [illegible] by hand and the mixture had a jelly-like consistency. The mixing was continued for 1 hour to obtain the microscopic state of disintegration, although a 15 minute [illegible] appeared to be sufficient for this purpose.

The viscosity of the mixture was found to be 10,400 cps. and its solids content was [illegible] 8.05% by weight.

This mixture was then employed as a binder, being pumped through a filter to storage tanks and subsequently sprayed on tobacco by the method and equipment described in U.S. Patent 2,734,513. It was applied at the rate of 3 grams per square foot to form a reconstituted tobacco sheet having the following physical properties:

Basis weight, gms./ft.² (1)	10.2
Moisture content (percent by weight)	13.0
Tensile, kg./in.	0.82
Fold tensile, kg./in.	0.42
Work-to-break, gm. cm./ft.² (2)	16.0

(1) Weight of all the ingredients, including tobacco pulp, reasonably free of moisture.
(2) Computed by an integrator attached to the Instron Tensile Tester.

Example 17

Burley tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then dried and ground and used to make a binder as follows.

One hundred parts of water were brought to a temperature of 195° C., and to this were added:

7.00 parts by weight (dry basis) of the washed burley stems,
1.05 parts diammonium phosphate, and
0.70 part of glycerin, as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 7.1 but no higher than 9.0.

The mixture was then stirred for one hour and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 18

Burley tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then used directly in the wet condition to make a binder as follows:

One hundred parts of water were brought to a temperature of 195° C., and to this were added:

7.00 parts by weight (dry basis) of the washed burley stems,
1.05 parts diammonium phosphate, and
0.70 part of a glycerin, as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 7.1 but no higher than 9.0.

The mixture was then stirred for one hour and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 19

Burley tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then dried and ground and used to make a binder as follows:

One hundred parts of water were brought to a temperature of 195° C., and to this were added:

7.00 parts by weight (dry basis) of the washed burley stems,
1.05 parts diammonium phosphate, and
0.70 part of triethylene glycol (TEG), as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 7.1 but no higher than 9.0.

The mixture was then stirred for one hour and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

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Example 20

Burley tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then used directly in the wet condition to make a binder as follows:

One hundred parts of water were brought to a temperature of 195° C., and to this were added:

7.00 parts by weight (dry basis) of the washed burley tobacco stems,
1.05 parts diammonium phosphate, and
0.70 part of triethylene glycol (TEG), as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 7.1 but no higher than 9.0.

The mixture was then stirred for one hour and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 21

Bright tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then dried and ground and used to make a binder as follows:

One hundred parts of water were brought to a temperature of 100° C., and to this were added:

7.00 parts by weight (dry basis) of the washed bright (flue cured) stems,
1.05 parts diammonium phosphate, and
0.70 part of glycerin, as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 8.0 but no higher than 8.5.

The mixture was then stirred for four hours and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 22

Bright tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then used directly in the wet condition to make a binder as follows:

One hundred parts of water were brought to a temperature of 100° C., and to this were added:

7.00 parts by weight (dry basis) of the washed bright (flue cured) stems,
1.05 parts diammonium phosphate, and
0.70 part of glycerin, as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 8.0 but no higher than 8.5.

The mixture was then stirred for four hours and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 23

Bright tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then dried and ground and used to make a binder as follows:

One hundred parts of water were brought to a temperature of 100° C., and to this were added:

7.00 parts by weight (dry basis) of the washed bright (flue cured) stems,
1.05 parts diammonium phosphate, and
0.70 part of triethylene glycol (TEG), as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 8.0 but no higher than 8.5.

The mixture was then stirred for four hours and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 24

Bright tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then used directly in the wet condition to make a binder as follows:

One hundred parts of water were brought to a temperature of 100° C., and to this were added:

7.00 parts by weight (dry basis) of the washed bright (flue cured) stems,
1.05 parts diammonium phosphate, and
0.70 part of triethylene glycol (TEG), as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 8.0 but no higher than 8.5.

The mixture was then stirred for four hours and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 25

In the acid wash treatment of tobacco parts for removal of alkaline earth minerals of the tobacco pectin, it is necessary to use quite large volumes of water if it is desired to reduce the soluble anion content of the product to a very low level. In this case, the use of a cation exchange resin in a closed loop system with the tobacco parts can make it possible to conduct the process with limited amounts of water and acid.

The use of a resin (such as Dowex 50 W) in this way does not at all alter the principles of this form of treatment since the resin merely serves as a convenient reservoir of acidity, continuously reconditioning effluent from the stems for reuse in the extraction.

In the following example, the use of a cation exchange resin permits the treatment of stems with a small fraction of their natural nitrate ion content. Hydrochloric acid is used at intervals to regenerate the resin, but never directly contacts the tobacco.

The apparatus used for ion-exchange extraction of calcium from tobacco stems consisted of a 20 inch diameter washing column fitted with a 20 mesh screen to support the charge of stems and a 6 inch diameter Pyrex column filled with 13 pounds of Dowex 50 W-8 acid-form ion exchange resin beads. The valves and piping permitted a pump to be used either for recycling waters from the stem charge through the column or for sep-

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acately regenerating the resin with hydrochloric acid to ab

In operation, fifty pounds of stems were placed in the washing column and rinsed with cold water until they were nearly free of water-soluble material. Then, the effluent waters were diverted through the ion exchange bed, returning by gravity to the top of stem charge. When the aqueous extract from one pound of burley stems was added to the recycling waters, a pH of between 1.5 and 2.0 was soon attained. During the treatment, the waters flowing from the resin column were monitored for their calcium content. (An appreciable precipitate from 50 ml. of resin effluent treated with ammonium oxalate and made basic with ammonia indicated that the resin was saturated with calcium and needed to be regenerated with hydrochloric acid.) After sixteen hours of operation, and one regeneration of the ion exchange resin, the treatment was considered complete. A subsequent analysis showed that the calcium content of the stem had been reduced to less than one-tenth of the starting value of 4.5% CaO. The stems were rinsed twice with cold distilled water, pressed lightly, and dried in a tobacco processing oven to a final moisture content of about 4%, and ground to pass a 40 mesh screen. When a small sample of this powder was stirred with a little cold ammonia, it dispersed immediately, forming a slip paste.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 26

Twenty grams of tobacco fines were washed in 1 liter of distilled water to get rid of the tobacco solubles. The fines were then admixed with 1 N hydrochloric acid until the mixture had a pH of 1.35. The pH was checked on a Beckman pH machine and the curves were also plotted against a titration of distilled water.

After titration, the acid was washed off the fines with three 400 ml. portions of distilled water. The fines were redispersed in 400 ml. of distilled water and sodium bicarbonate was added to bring the pH to 7. The slurry was cast at a thickness of 30–50 mils and dried to form a sheet and the resulting sheet was used as the adhesive binder in a sandwich-type reconstituted tobacco sheet.

Example 27

Twenty grams of tobacco fines were washed in 1 liter of distilled water to get rid of the tobacco solubles. The fines were then admixed with 1 N hydrochloric acid until the mixture had a pH of 1.35. The pH was checked on a Beckman pH machine and the curves were also plotted against a titration of distilled water.

After titration, the acid was washed off the fines with a 400 ml. portion of distilled water. The fines were redispersed in 400 ml. of distilled water and sodium bicarbonate was added to bring the pH to 7. The slurry thickened to a viscous mass which is evidence that the pectin had been solubilized. The slurry was cast at a thickness of 30–50 mils and dried to form a sheet and the resulting sheet was used as the adhesive binder in a sandwich-type reconstituted tobacco sheet.

Examples 28–30

In each of these three examples, 20 gms. of tobacco fines were washed in 1 liter of distilled water to get rid of the tobacco solubles. The fines were then admixed with 1 N hydrochloric acid until the mixture had a pH of 1.35. The pH was checked on a Beckman pH machine and the curves were also plotted against a titration of distilled water.

After titration, the acid was washed off the fines with three 100 ml. portions of distilled water. The fines were redispersed in 400 ml. of distilled water and a neutralization agent, as indicated in Table V, was added to bring the pH to the value shown in Table V. The slurry in each case thickened to a viscous mass which is evidence that the pectin had been solubilized. The slurry was cast at a thickness of 30–50 mils and dried to form sheets. The binders formed in this way were tested to determine their physical properties. These properties are shown in Table V below. The binder was used as the adhesive in a sandwich type reconstituted tobacco sheet.

TABLE V.—PROPERTIES OF BINDER FILM MADE BY ACID WASH PROCESS

Example	28	29	30
pH	7.1	7.1	7.1
Sheet wt., gm./ft.	4.36	4.79	4.73
Neutralization agent	(1)	(2)	(3)
Integrator count	884	884	884
Percent moisture	11.3	11.6	11.5
Percent elongation	1.68	1.68	1.68
Tensile, kg./in.	5.83	5.83	5.83
Tensile coefficient, kg./in. / gm./ft.	6.58	6.58	6.58
Work coefficient, gm. cm./sq. in.	6.48	6.48	6.48

(1) Conc. NH4OH.
(2) 10% KOH.
(3) 10% NaOH.

In the table the terms have the following meanings:

(1) *Integrator count* is proportional to the area under the stress-strain curve recorded by an Instron Test Machine.

(2) Percent moisture—The percentage of moisture contained in the entire sheet (wet basis).

(3) Percent elongation—The percentage elongation, at breakage, on an Instron Test Machine.

(4) Tensile, kg./in.—The breaking strength of a 10 cm. test strip which is 1 inch wide; an average of 10 strips.

(5) *Tensile coefficient*—Kg. per gm. of basis weight.

(6) Work coefficient, gm. cm./sq. in.—Work coefficient is proportional to the product of the tensile strength and the elongation.

Example 31

The following ingredients were mixed into 900 parts of hot water and heated for 3 hours at 85°–90° C., maintaining a pH of 7.0 by adding small amounts of aqueous ammonia.

	Parts
Mixed manufacturing byproducts and scrap tobacco	87.9
Diammonium ortho phosphate (DAP)	5.0
Glycerin	5.0
Corn syrup	2.0
Potassium sorbate	0.14

The slurry was then refined in a one gallon capacity Waring Blender for 5 minutes. With this amount of refining, the fibrous portion of the composition was seen to be well dispersed and thoroughly interlocked when examined in a droplet at a 1:9 dilution. This examination was facilitated by the addition of a small amount of Congo red dye.

The composition was cast on stainless steel panels with an eight inch angle knife set for wet film thicknesses of 0.020 to 0.045 inch, and dried on a steam table.

The wet strength of the films was determined on a Scott Serigraph IP-2 using 1 inch width strips. The center of each strip was wet with distilled water on both sides and allowed to become thoroughly wet through (30 seconds or one minute) before actuating the tensile mechanism. These films showed the extraordinarily high wet film strength of 140 grams at a film weight of 9.5 grams per square foot. A high wet strength is highly desirable in the preparation of cigarette filler, and assures that the subsequent manufacturing performance of this material will exceed that of the finest grades of leaf tobacco. Such strength is uniquely obtainable in the presence of the liberated form of pectin described in this disclosure.

We claim:

1. A process for producing reconstituted tobacco sheet material which comprises contacting comminuted tobacco parts with a treating solution containing a reagent comprising ammonium orthophosphate so as to destroy the alkaline earth metal cross-links which hold the pectinaceous material and thus occasioning release of the pectins

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by the formation of a precipitate comprising an insoluble calcium or magnesium salt, and finally casting the tobacco and the in situ formed pectins to form a reconstituted sheet, the pectins serving as a binder for the treated tobacco parts in the final sheet.

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MELVIN D. REIN, *Primary Examiner.*

U.S. Cl. X.R.

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512-52, 010

United States Patent Office

3,353,541
Patented Nov. 21, 1967

3,353,541
TOBACCO SHEET MATERIAL
John D. Hind and Robert B. Seligman, Richmond, Va., assignors to Philip Morris Incorporated, New York, N.Y., a corporation of Virginia
No Drawing. Filed June 16, 1966, Ser. No. 557,983
1 Claim. (Cl. 131—17)

ABSTRACT OF THE DISCLOSURE

This disclosure relates to a process for producing a binder composition for use in the manufacture of reconstituted tobacco. The binder is made from tobacco plant parts and involves the use of the naturally occurring tobacco pectins, which are obtained by a process in which diammonium acid phosphate is employed to treat the tobacco plant parts. The treatment involves the destruction of the alkaline earth metal cross-links of the tobacco pectins, the release of the resulting tobacco pectins by a swelling action and the depositing of the released tobacco pectins on the treated plant parts.

This application is a continuation-in-part of application, Ser. No. 336,009, which was filed on Jan. 6, 1964, now abandoned, and which, in turn, is a continuation-in-part of application, Ser. No. 240,130, filed Nov. 26, 1962, now abandoned, and application, Ser. No. 169,995, filed Jan. 26, 1962, now abandoned.

This invention relates, generally, to tobacco compositions and methods of producing such compositions. More particularly, the invention relates to improved adhesive materials as binders for reconstituted tobacco compositions. In addition, the invention relates to methods for the production of novel adhesive compositions comprising tobacco pectins, to reconstituted tobacco containing tobacco pectins as binders, and to smoking compositions made therefrom.

During the production and processing of tobacco products, including aging, blending, sheet forming, cutting, drying, cooling, screening, shaping and packaging, considerable amounts of tobacco fines and tobacco dust are produced. It is known that such tobacco fines and dust can be combined with a binder to form a coherent sheet, which resembles leaf tobacco and which is commonly referred to as reconstituted tobacco. One method for making reconstituted tobacco of this general character is disclosed in United States Patent No. 2,734,510, wherein the tobacco fines and dust are applied to a binder made of carbaymethyl cellulose, carboxymethyl hydroxyethyl cellulose or a suitable salt thereof. The binder, in such compositions, ranges from about 5% to about 50% of the weight of the tobacco employed. United States Patent No. 2,708,173, describes a binder for reconstituted tobacco which consists of a plant gum, principally of galactomannan. United States Patent 2,592,554 to Frankenburg describes, as binders for reconstituted tobacco, various water-soluble polysaccharides, such as alginic and pectinic acids and their sodium and potassium salts, derived from plants other than tobacco, for example derived from citrus fruits. However, the addition of cellulosic binders further increases the amount of cellulosic material in the product and tends to create an acrid and bitter smoke, when the product is used to make cigarettes. The natural hydrophilic colloid gums such as guar gum, locust bean gum, algin and other commonly used materials, such as Irish moss, have additional disadvantages. These materials contain proteins and other materials not found in tobacco which add distinctive flavors of their own to tobacco products during smoking. Thus, Frankenburg, in describing the use of various water-soluble polysaccharides derived from plants other than tobacco, teaches that care should be exercised that they must be in a state of refinement. Frankenburg teaches that these materials should be free of extraneous matter containing compounds of nitrogen, particularly proteins, and compounds of sulfur, phosphorus and the halogens; i.e., compounds giving undesirable products of combustion or dry distillation. Such refining is often a very tedious and difficult operation.

The present invention makes possible the production of improved reconstituted tobacco by a method which is simpler and more effective than the methods previously employed. The present method does not require refining of the binder and is, therefore, more easily and efficiently employed than other methods for making binders and for making reconstituted tobacco. The reconstituted tobacco which is obtained in accordance with the present invention need not contain any additional cellulose or proteinaceous material which is foreign to tobacco, since the binder which is employed may be derived solely from tobacco, and may be produced in such a manner that it contains no materials other than those which naturally occur in tobacco. Thus, reconstituted tobacco produced in accordance with the invention, can be so formulated as to be similar in physical properties and chemical composition to natural tobacco.

The term pectic substances [1] will mean those substances which are found in many plant products, and which consist essentially of partially methylated galacturonic acids joined in long chains.

The pectic substances found in tobacco plants contain acetyl groups and differ considerably from commercially available pectins found in other plants, including sugar beet pectins and citrus and fruit pectins. Tobacco protopectins are uniquely insoluble in hot water as compared with protopectins from many other sources and comprise mainly water-insoluble pectins (protopectins) consisting of the calcium and magnesium salts of partially esterified and slightly acetylated polymers of galacturonic acid. The divalent calcium and/or magnesium atoms act as cross-links between acid chains, thus making the polymers water-insoluble. As an illustration, the structure of the

[1] Note: Unless otherwise specified, the term "pectin" will for convenience, hereinafter be employed interchangeably with the term "pectic substance."

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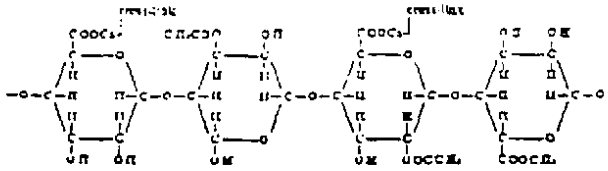
calcium salt of a polymer of galacturonic acid can be represented as follows:



Although pectins have long been known as constituents of plant tissue, it has been found extremely difficult to separate pectins from the remainder of plant compositions and to obtain them as homogeneous compositions. The recovery of pectins from tobacco is even more difficult than the recovery of pectins from other plants.

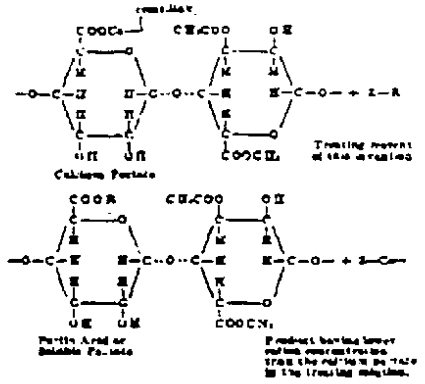
In accordance with the present invention, tobacco parts are bonded together by tobacco pectins which are specially prepared by a novel process which yields these pectins in a form in which they can be employed as binder materials. Our process for preparing tobacco pectins comprises first reacting tobacco parts, preferably in a form in which they present a large surface area, with an aqueous solution of a non-toxic reagent which is capable of reacting with and destroying the calcium and magnesium crosslinks in the pectinaceous substances which naturally occur in tobacco. After the calcium and magnesium crosslinks are destroyed, the tobacco pectins are liberated and made available for use as a binder. The tobacco pectins are then dissolved or dispersed in solution, or are at least sufficiently released from the interstices of the tobacco mass so that they form a coating on the surface thereof. Tobacco pectins which are dissolved or dispersed in the treating solutions, are thereafter precipitated or deposited from the solution, so that they become available for use as a binder material. In this way, the tobacco parts can be bonded together by a binder material which is made of ingredients that are closely related to the naturally occurring ingredients of tobacco. The bonding can be accomplished without the need for the purification of the tobacco pectins, inasmuch as any materials which are present are closely related to the materials which are normally present in tobacco and, thus, do not add any undesired qualities to the tobacco.

The tobacco parts which can be employed in the present invention including tobacco leaves, stems and stalks, or mixtures of these, whether in sheet, flake, particulate or other form. Preferably, the parts are ground, cut or otherwise prepared in a form which presents a large surface area. The portions of the plant comprising the stems or midribs, and often referred to as tobacco petioles, are the preferred starting materials. Tobacco stalks contain lesser amounts of pectinaceous materials but can also be employed.

In the first step of the process of our invention, tobacco pectins are liberated from pectinaceous materials in tobacco by reacting the pectinaceous materials with a reagent which, under the conditions of the reaction, is reactive with the calcium (and/or magnesium) contained therein to form a compound or product having a lower calcium ion, and, in the case of magnesium, magnesium ion, concentration[1] in the treating solution than the naturally occurring calcium (or magnesium) pectate. This reagent may, for convenience, be hereinafter referred to as "cross-link destroying reagent."

The reaction may be generally represented by Equation 1, which illustrates the reaction of one type of tobacco protopectin (a calcium salt of a polymer of galacturonic acid), wherein calcium crosslinks are present with the reagent of the present invention, in the equation,

R may be hydrogen, in which case the product is pectic acid, or R may be a monovalent inorganic cation, such as sodium, potassium or ammonium, in which case the product is a soluble pectate.



In one embodiment of this invention, the reagent, which can be, and preferably is, in aqueous solution, acts by forming a precipitate with the calcium or magnesium, in which case, it can be a water-soluble monovalent metal salt of the formula M_nX wherein M is a monovalent inorganic cation, n is an integer having a value of 1, 2, or 3 and X is an anion which may be monovalent or polyvalent, such that the calcium salt of the formula Ca_pX_q is essentially insoluble in the treating solution and p and q are integers corresponding to the functionality of X. Monovalent cations which are effective include the alkali metals such as sodium, potassium and lithium, and also include such monovalent cations as ammonium, and substituted ammonium ions $(NR_4)^+$, where R=aryl or alkyl. The anion portion of the molecule may be $CO_3^{=}$, $PO_4^{\equiv}$, $HPO_4^{=}$, $H_2PO_4^{-}$, and the like. For example, the compound MnX could be sodium carbonate, Na_2CO_3, since sodium is a monovalent inorganic cation and calcium carbonate is essentially water insoluble. Additional representative examples of precipitating agents are the orthophosphates, metaphosphates and carbonates of sodium, potassium, lithium and ammonium. In the case of the orthophosphates, the anion portion of the molecule may be either $PO_4^{\equiv}$, $HPO_4^{=}$, or $H_2PO_4^{-}$. Specifically, for example, when ammonium orthophosphate is used, the precipitate is calcium and/or magnesium ammonium phosphate. The pH of this reaction should be between about 5.8 and 10 and the temperature may be as high as 400° C. but should, preferably, be between about 25° C. and about 135° C. for a period of from about 1 minute to about 24 hours. Preferred precipitating agents which may be employed are the alkali metal carbonates, for example,

[1] By concentration or mass concentration or activity as set forth in Glasstone, "Textbook of Physical Chemistry," 2nd edition, p. 931, D. V. Nostrand Company, Inc.

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sodium carbonate and potassium carbonate. Particularly preferred precipitating agents which may be employed are the alkali metal phosphates and, most particularly, the alkali metal orthophosphates and the ammonium orthophosphates, such as the ammonium phosphates and ammonium orthophosphate, sodium orthophosphate, potassium orthophosphate, sodium dihydrogen orthophosphate, ammonium dihydrogen orthophosphate, potassium dihydrogen orthophosphate, diammonium monohydrogen orthophosphate, disodium monohydrogen orthophosphate and dipotassium monohydrogen orthophosphate.

In a second embodiment of this invention, the cross-link destroying reagent acts by sequestering the calcium or magnesium, thereby removing the calcium or magnesium atoms by forming a complex therewith. Suitable reagents of this type include any sequestering agent which will form a complex or chelate with the calcium and/or magnesium, thereby removing the calcium and/or magnesium and making them unavailable for recross-linking with the pectin. Illustrative of such sequestering agents are ethylene-diaminetetraacetic acid and similar amino acids, alkali metal polymetaphosphates such as tetra-metaphosphates, hexametaphosphates and trimetaphosphates, pyrophosphates and tripolyphosphates, such as sodium hexametaphosphate, tetrasodium pyrophosphate and pentasodium tripolyphosphate. The mechanism which occurs when a sequestering agent is employed is the formation of a chelate; calcium and magnesium ions are no longer available to combine with the pectate ions in solutions. Many naturally occurring amines and peptides are also effective as sequestering agents for calcium and/or magnesium. Representative examples include alanine, aspartic acid, glycine, glycyl glycine, glutamic acid, serine, tyrosine and diiodo-l-tyrosine. Amino acids that are effective as chelating solubilizing agents include beta alanine, N,N-diacetic acid; amino barbituric acid, N,N-diacetic acid; 2-amino-benzoic acid, N,N-diacetic acid; beta-aminoethylphosphonic acid, N,N-diacetic acid; beta-aminoethylsulfinic acid, N,N-diacetic acid and ethylene-diamine-tetraacetic acid. The pH of this reaction should, preferably, be between about 4 and about 10 and the temperature should, preferably be between about 0° C. and about 145° C. for a period of from about 1 minute to about 24 hours.

A cross-link destroying reagent may also function partially as a precipitating reagent, in accordance with the first embodiment of this invention, and partially as a sequestering agent, in accordance with the second embodiment of this invention. Such a reagent, for example, is diammonium monohydrogen orthophosphate (DAP), which is a particularly preferred material, in accordance with the invention.

In a third embodiment of this invention, the cross-link destroying reagent is an acid wash which forms the released but insoluble free pectic acid and soluble calcium and magnesium salts. Generally the acid wash will comprise an inorganic acid, such as hydrochloric acid, phosphoric acid, sulfuric acid or a similar acid, which will form soluble calcium and magnesium salts under the following conditions. Hydrochloric acid and sulfuric acid are particularly preferred. The acid may be employed as 0.25 N to 3.0 N solutions, but is preferably employed as 0.5 N to 1.5 N solutions. The exact dilution and amount to be employed will vary with the particular acid which is used, it only being necessary that sufficient acid be present to convert the calcium and magnesium present in the tobacco being treated to the calcium and magnesium salts of the acid. The acid treatment is preferably conducted at a temperature of from about —1° C. to about 50° C. The acid treatment comprises reacting the tobacco parts with the acid until the resulting mixture has a pH of from about 1.0 to about 2.5. Preferably, the pH is brought to from about 1.0 to about 1.7, the most desirable pH being between 1.15 and 1.55. This treatment will generally be conducted from about 10 minutes to 24 hours, depending in part on the size of the tobacco particles. The acid conditions which are necessary for this embodiment of the invention may be achieved by the use of ion exchange resins which may be used, with suitable recycle to obtain the desired pH of the solution during treatment. The ion exchange resins may be used and regenerated in accordance with the usual practices for such resins.

Preferably, the mixture resulting from the acid treatment is then washed with water. This water wash step is preferably conducted at a temperature of from about 15° to about 35° C. and, preferably, distilled water is employed. When this wash step is employed, sufficient water should be used to remove the calcium and magnesium salts of the acids, which salts are formed in the above-described treatment; thus, there should be at least 2 volumes of water per volume of the mixture resulting from the acid treatment. The wash water is separated from the tobacco by any suitable means, for example, by conducting the wash to a centrifuge, filter press, Buchner funnel, or any other apparatus from which liquids can be substantially removed from solid materials.

In summary, in the first step of the first embodiment of this invention the treating agent Z-R attacks the calcium and/or magnesium cross-links of tobacco protopectin and forms a precipitate which is a salt of calcium and/or magnesium, thus removing the calcium and/or magnesium from the protopectin and from the solution. In the first step of the second embodiment, the treating agent Z-R is a sequestering agent which forms a chelate of the magnesium and/or calcium from the tobacco protopectin and makes the calcium and/or magnesium unavailable for recombining with the pectins. With certain reagents, such as DAP, the first step of the present process may comprise a combination of the mechanisms of the first embodiment of this invention and the mechanism of the second embodiment of this invention. In the first step of the third embodiment of this invention, the treating agent Z-R is an acid which attacks the calcium and/or magnesium cross-links of the tobacco protopectin and forms the soluble calcium and/or magnesium salts, which are then washed away from contact with the pectins.

In the first step of each of the first two embodiments of the invention, the pectin which results is in condition for release from the tobacco cell structure. R in Equation I being a monovalent inorganic cation such as sodium. In the first step of the third embodiment, the insoluble pectic acid resulting from the acid treatment must be reacted with an alkaline material before it is in condition for release from the tobacco cell structure.

The acid treated pectins are placed in condition for release by bringing the mixture resulting from the acid treatment, and preferably, after the water wash described above, to a pH of from about 5.0 to about 10.5 and, preferably, from about 6.1 to about 8.5, by the addition of an alkaline material. Suitable alkaline materials include ammonium hydroxide and alkali metal hydroxides, for example, sodium hydroxide, potassium hydroxide and lithium hydroxide, and alkali metal salts, such as sodium bicarbonate, sodium carbonate, sodium phosphate, and similar salts to convert the pectic acid to a soluble form. The alkaline material may be any water-soluble compound containing a monovalent inorganic cation and capable of producing hydroxide ions when dissolved in water. The temperature of this step may be from about —1° C. to about 45° C., but is, preferably, from about 15 to about 35° C. The alkaline material is preferably in the form of solid particles or in the form of a solution having a concentration of from about 5 to about 30%.

Once the tobacco pectins have been liberated from the tobacco, by the removal of the calcium and magnesium cross-links, they should be released from the interstices of the tobacco. That is, they will be made available to the

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solution or suspension or, in certain instances, they will be merely deposited on the surface of the tobacco particles. This comprises the second step of the process of the present invention. In embodiments 1 and 2 of the invention, this release or second step may be accomplished concurrently with the first step by reacting with the solution of the treating reagent. In embodiment 3, however, as indicated above, the insoluble pectic acid resulting from the treatment should be reacted with an alkaline material before it can be released. In such event, the release may be concurrent with the addition of the alkaline material due to a washing action. In any case, additional treating liquid or water may be used to effect the release through a washing action of the treated tobacco particles.

In accordance with the third step of our process, the liberated and separated tobacco pectins can next be precipitated or deposited in a relatively *free form* (as compared with the tobacco pectins as they were originally present in the tobacco), from the treating solution, for example, by being formed into the insoluble pectic acid or into an insoluble salt of pectic acid or by the action of a water-miscible solvent, such as acetone or ethyl alcohol to cause a water-soluble salt of pectic acid to go out of solution. In the case of tobacco pectin solutions such as solutions of sodium and potassium pectinates and/or pectates, this can be accomplished by acidifying the solution until the pectins precipitate or by adding a gelation agent, such as an alcoholic solution, *preferably* having a pH of *from* about 1 to 9 and, *preferably*, *from* about 1 to 3. The pH of the alcoholic solution can be regulated by the addition of a mineral acid, such as HCl, to the alcohol. Although the preferred gelation agent is ethanol, any water-miscible organic solvent having up to about 10 carbon atoms may be employed, for example, a ketone, such as acetone, or a diether, such as dioxane. Water-immiscible solvents such as ether, for example, ethyl ether, can be used, if combined with a water-miscible solvent, such as acetone.

The tobacco pectins can be recovered by concentrating the solution or suspension in which they are present until they precipitate. This precipitate might also be characterized as an extractable mass, since the pectin solution, upon concentration, generally becomes progressively more viscous until it finally dries to leave a deposit in a glassy solid state.

While the tobacco pectins can be separated and purified before use as a binder in reconstituted tobacco sheets, they are preferably employed just as they are produced *in situ*, i.e., in combination with the treated tobacco plant parts from which they were obtained, the entire combination comprising the binder for reconstituted tobacco sheets or, under some circumstances, the entire combination comprising essentially the entire components of a reconstituted tobacco sheet. By using the entire mixture, no original tobacco flavors are lost, all of the tobacco is employed, and no expensive and time-consuming refining operations are required.

Although it is not necessary, the thixotropic properties of solutions containing soluble pectins can be adjusted in the preparation of a cured sheet by the addition of such materials as calcium chloride. If any complex or precipitate formed in the first step of this process is present with the soluble pectates, the thixotropic properties of the mixture can also be adjusted by adjusting the pH to precipitate calcium and magnesium pectates.

A preferred preliminary step, in accordance with the present invention, comprises washing the tobacco plant parts, which are preferably ground or cut to a relatively small size, with cold water. This water wash serves to remove impurities which might otherwise hinder the subsequent treatments in accordance with the present invention. It is particularly desirable to employ such a cold water washing step when alkali metal carbonates are employed as the reagent in the first step. Generally, sufficient water should be used during such a water wash

operation to cover all of the tobacco plant parts present. The temperature of the water may be between −1° C. and 100° C., but is preferably about 20° C., and the water wash should generally continue for a period of from about ½ to 2½ hours. Agitation during the wash is desirable, but not necessary. After the water wash has been completed, the water can be removed from the tobacco parts by filtration, decantation or other suitable means.

As discussed above, a particularly preferred embodiment of the present invention involves the use of an ammonium or alkali metal orthophosphate, such as diammonium monohydrogen orthophosphate (DAP), for the release of the tobacco pectins. The DAP will, generally, be added to the tobacco plant parts, which may, for example, be bright tobacco parts, burley tobacco parts, or a mixture of the same, in an aqueous solution. The concentration of the DAP in the aqueous solution is not critical, but will, generally, be in the range of 0.5–5.0% by weight. The DAP and water may be added separately to the tobacco. The amount of DAP should, preferably, comprise *from* about 0.01 to about 0.5 part, and, most *preferably*, *from* about 0.05 to 0.35 part (by weight) per part of tobacco being contacted. A humectant, such as glycerin or triethylene glycol, may be present, if desired, at about 0.5 to 1 part per part of DAP. The temperature during the DAP treatment of the tobacco may vary between room temperature and about 150° F. or higher, depending on the type of tobacco being treated. Under pressure, even higher temperatures may be employed. The pH of the mixture is, preferably, maintained at a value about 7.1 to 9.0, which may conveniently be accomplished by the addition to the solution of concentrated aqueous ammonia. By agitating or stirring such a mixture under the above-described conditions for from about 1 minute to about 1 day, and *preferably*, from about 1 hour to about 3 hours, the tobacco pectins are liberated, released and deposited in the tobacco plant parts to form a binder composition. The resulting mixture may then be refined, for example, in a disk refiner, until substantially all the pulp (in excess of the water present), can be shaken through a screen of approximately 18 mesh to produce a binder composition which is ready for use in the manufacture of reconstituted tobacco sheets.

The term "tobacco pectins" as used throughout this specification means "liberated tobacco pectins" and comprehends pectins which have been freed or liberated from tobacco and are, therefore, not bound into the tobacco structure, as differentiated from the insoluble, naturally-occurring protopectins which are bound into a plant cell structure. The term includes the free pectinic or pectic acid, as well as soluble salts such as the sodium, potassium, ammonium, pectates and pectinates, and insoluble salts such as the calcium and magnesium pectates and pectinates depending on what method is employed to liberate and obtain them from the naturally occurring insoluble protopectins.

The tobacco pectins produced or liberated *in situ* or isolated by means of this invention can be used as the sole binder material for reconstituted tobacco, i.e., no other materials need be added to make the sheet. They can be sprayed, extruded or cast, thus facilitating application onto a moving belt carrying tobacco dust. Under proper conditions of formulation and processing, reconstituted tobacco made with the tobacco pectins produced by this invention exhibit excellent physical and aromatic properties. The ultimate tensile and wet strengths of the reconstituted tobacco are good. While no other materials need be added to the pectinaceous binder, other materials can be added, if desired. For example, organic acids and preservatives which may in themselves be of tobacco origin, may be added. Plasticizers, such as glycols and polyglycols, and humectants, such as glycerin, may also be added, if desired. In addition, the gel strength of the tobacco pectins can be regulated by partial precipitation

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to control such rheological properties as viscosity, fluidity and elasticity. Other additives or dispersants may be added in small amounts to regulate slurry qualities, provided, however, that such substances are not added in large enough quantities to adversely affect the flavor or aroma of the final product. Furthermore, the tobacco pectins can be combined with water-soluble gums or water dispersible gums commonly used as binders for tobacco sheets such as methyl cellulose, sodium carboxymethyl cellulose, guar gum, locust bean gum, or alginates, although it is preferred to minimize or eliminate such additions in order to obtain a product which most closely resembles natural tobacco.

The product from treating the tobacco plant parts in accordance with the methods of the present invention may be cast directly and dried and cut into particulate material similar in physical form to ordinary smoking tobacco and so used, preferably mixed with tobacco leaf cut or shredded in the usual manner. The product may be cast in sheet form, in blocks or as threads or other shapes, as desired. An important use, however, of the prepared composite slurry or easily molded isolated pectinaceous mass is as a binder for ground tobacco and for the making of corresponding tobacco products suitable for smoking. Sheet material of widely different properties may be formed by suitable variations in the manner of forming. One method and product comprises flowing the composite slurry onto a moving belt and applying a layer of dry ground or fragmented tobacco to the wet adhesive surface. If desired, there may be first applied to the belt a layer of the tobacco, followed by a layer of the binder, and then a top layer of the tobacco. Various additives may be included with the ground tobacco such as flavorants, plasticizers and aromatic substances. The web is ultimately dried and then suitably moistened and rolled up. Such methods of forming continuous sheets are known generally in the art and the details need not be further described. Representative of this procedure is the apparatus and method disclosed in U.S. Patent 2,734,513.

Another method of forming a reconstituted tobacco product, with the slurry of the isolated tobacco pectins as a binder, comprises mixing ground tobacco thoroughly therewith into a mass of dough-like consistency and then casting the mass in sheet form onto a moving belt surface followed by drying and remoistening in accordance with the known procedures. Representative of this procedure is the apparatus and method disclosed in U.S. Patents 2,708,-35 and 2,769,734. Obviously, the reconstituted tobacco may also be formed by molding or other suitable means.

A particularly preferred aspect of the present invention comprises employing, as a binder or directly, the mixture of tobacco and tobacco pectins which have been produced in situ, without any separation steps and without the necessity for any additional adhesive materials.

The following examples are illustrative:

Example 1

Tobacco stems (10 parts) were covered with cold water and leached for ½ hour. The water was then decanted and discarded. A treating solution, made from 1 part of sodium carbonate dissolved in 60 parts of water, was added to the leached parts. This mixture was heated at its boiling point for 30 minutes at atmospheric pressure and then for 20 minutes at 20 p.s.i.g. In the course of the treatment, the tobacco pectins were dissolved from the tobacco parts. The entire wet mass (pulp mixture) was dried and ground in a Waring Blendor so that it would pass through a 50 mesh screen. The resultant material had gel-like properties and was thixotropic in nature.

To this material was added 2 parts of glycerin, to serve as a humectant. The pH of the resulting mixture was adjusted to 6 by the addition of a solution of 10% hydrochloric acid. One gram of sodium carboxymethyl cellulose (CMC) was added to the mixture, giving a proportion of about 1 part of CMC to 10 parts of total solids in the mixture. Since it was desired to use this mixture as a sprayed tobacco binder, the CMC was employed in order to adjust the spraying quantities. The mixture of (a) treated tobacco plant parts, including the liberated tobacco pectins, (b) the sodium carbonate, (c) the glycerin and (d) the CMC had a viscosity which was suitable for spray application of the mixture as a binding material for reconstituted tobacco. This material was sprayed on top of an undercoating of tobacco dust, of an 80 mesh size, which had been dusted on a wet belt. Another coat of the tobacco dust was laid on top of the binder. In general an apparatus similar to that described in U.S. Patent 2,734,513 was employed. As in said patent, the reconstituted sheet was dried and was then humidified to the desired moisture content. The control in Table I was made in a conventional manner, using a relatively large amount of CMC in proportion to the pulped washed tobacco stems. The test data as observed are recorded in Table I.

TABLE I

Sample	Sheet 1	Sheet 2	Sheet 3	Control
Part CMC [1]	1/10	1/10	1/20	1/2.3
Moisture (percent) [2]	12.3	12.1	12.7	11.8
Tensile, lbs./in. [3]	.80	.90	.88	.72
Tobacco Texture, scale [4]	7.1	7.0	6.1	5.2
Elongation (percent) [5]	5.1	5.3	5.0	4.9
Burning Rate, sq./min. [6]	1.8	1.8	1.9	1.7

[1] Part CMC per parts of total solvent-water in the slurry (including tobacco parts).
[2] The percentage of moisture contained in the entire sheet (wet basis).
[3] The breaking strength of a 10 mm. wide strip of sheet when subjected to a tensile force at 20 cm/min.
[4] Open bulk, soft and full textured by subtraction of a 600 grams weight for 30 seconds.
[5] The percent elongation, at breaking point, on the Instron Test Machine.
[6] Time (in minutes) burning rate of a single test strip, in air.

The reconstituted tobacco sheets made pursuant to this example were shredded and made into cigarettes. The test cigarettes, as well as control cigarettes made from standard reconstituted tobacco sheet, were submitted to a smoking panel for subjective testing. The panel found that the smoke of the cigarettes made by the method of this invention was significantly less harsh than the smoke obtained from the control cigarettes. A pleasing vanillin odor was found to be transmitted into the aerosol phase of the smoke from the test cigarettes made employing reconstituted tobacco made by the present invention.

Example 2

Fifty grams by weight of burley tobacco stems were thoroughly washed in about 5 liters of cold water for three hours. The washed tobacco stems were then mixed with 500 grams of water having dissolved therein 5 grams of sodium carbonate. The resultant mixture, containing about 8% solids, was steamed under atmospheric pressure for 30 minutes and under a pressure of 20 p.s.i.g. for an additional 20 minutes. At the end of this time, the mixture was allowed to cool and the liquid was separated from the solid materials. The solid materials were treated in a cider press to recover as much as possible of the remaining liquid. The liquid was added to a previously prepared coagulant bath which consisted of ethanol and hydrochloric acid, in an amount to adjust the pH of the coagulant to about 1.0. The resulting mixture (which had a pH of about 3.0) was alternately stirred and allowed to settle for a period of two hours. At the end of this time the mixture was strained through a cloth sieve and the filtrate was discarded leaving a solid, gel-like mass, consisting essentially of pectinaceous materials combined with approximately 10 parts of liquor. The yield of pectinaceous materials was 15%, based upon the dry weight of the starting tobacco plant parts. The mass was observed

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to be thixotropic, soluble in water at a pH of about 6 and soluble in a sodium carbonate solution.

The tobacco pectins thus isolated were quite impure and had a color characteristic of tobacco. The solid mass was then redissolved in a sodium carbonate solution and the resultant solution was poured into an acidified ethanol bath similar to the coagulant bath used earlier. The recoagulated solid was obtained by filtration and dried in an oven at 105° C. The dried tobacco pectins were in the form of an opaque, substantially colorless sheet. When the sheet was pulverized, a white powder was obtained having a distinct and pleasing odor similar to vanillin.

Ten grams of the dried tobacco pectins prepared above were swelled in 100 ml. of cold water. The mixture was then added to a slurry consisting of 8.5 grams of refined kraft pulp dispersed in 200 ml. of water. The resultant mixture was homogenized in a Waring Blender. Four grams of glycerin were then added to the mixture to serve as a humectant. The resulting mixture was cast into a binder film, using a Gardner casting knife set to produce a sheet having a wet thickness of 50 mils. The physical properties of this sheet, after drying, were tested and were found comparable to the properties of conventional binder films. The physical test data are given in Table II.

The control material was made by mixing pulp, which was beaten refined with NaOH, with sodium carboxymethyl cellulose (CMC) and a humectant, as is shown in Table II.

TABLE II

	Test Binder-Film	Control Binder-Film
Binder Material	(*)	(*)
Basis Weight, grs./ft.²	4.3	4.44
Moisture, Percent	14.3	14.9
Opacity Coefficient	0.88	0.87
Tensile Tensile, g./in.	4.9	4.3
Elongation, Percent	4.1	4.1
Tear Coefficient, grams cm.-in. in.⁻¹	37.88	41.9

* Dried tobacco pectins extracted from tobacco particles.
* CMC—Pulp—CMC (sodium carboxymethyl cellulose) and pulp, prepared by above methods and containing about 30% CMC.
* Tear coefficient is proportional to the product of the tensile strength and the elongation.

Example 3

One hundred grams of burley tobacco stems were covered with distilled water, leached for 30 minutes and then drained. The washed stems were then mixed with a solution containing 10 grams of diammonium monohydrogen orthophosphate in 600 ml. of water. The resultant slurry was heated for 1 hour at a temperature of from 90 to 100° C. The pH of the reaction mixture, after completion of the reaction, was about 7. The entire reaction mixture was homogenized in a Waring Blender. The solids content was determined to be 5% by weight.

Two grams of glycerin, to serve as a humectant, were blended with 200 grams of the above reaction mixture. A film of 50 mil wet-thickness was cast from this mixture. The extruded film was found to have the property of being able to slide off a surface even when newly cast. After the film was partially dried, it was found that it could be peeled off a plate by hand, while still partially wet and then hung up to dry like wet cloth. This property provides definite advantages in the manufacture of tobacco products.

Physical tests were carried out on the dried film thus prepared and were also carried out on a control film made by using conventionally pulped tobacco plant parts with sodium carboxymethyl cellulose (CMC) as the binder. A film of binder as made in this example was burned and was found to give a pleasant aromatic smoke. The test data are given in Table III.

TABLE III

	Test Binder-Film	Control Binder-Film
Binder Material	(*)	(*)
Basis Weight, grs./ft.²	4.5	4.6
Moisture, Percent	14.7	14.3
Tensile Coefficient (*)	4.13	4.47
Tensile Tensile, g./in.	2.8	3.3
Elongation, Percent	13.1	4.1
Tear Coefficient, grs. cm./in. in.⁻¹	36.9	37.9

* Based on initial conditions.
* Tobacco pectins not formed in situ.
* CMC+Pulp.
* Lbs. per sq. in. of basis weight.
* Tensile, lbs. per 1/2 inch, load basis.
* Tensile coefficient is proportional to the product of the tensile strength and the elongation.

Examples 4–10

In each of these examples, burley tobacco stems were covered with distilled water, leached for 30 minutes, and drained. One hundred grams of stems were treated with the agent indicated in Table IV. The agent was dissolved in 600 ml. of distilled water in an amount sufficient to make up a 10% (by weight) solution. The leached tobacco stems were placed in said solution and heated in a steam bath for 1 hour at a temperature of from 90 to 100° C. (Except for the example in which Versene was employed. That example was conducted at room temperature.) A five ml. aliquot was taken from the resulting mixture and mixed with 20 mls. of ethyl alcohol in a graduated cylinder whereby a precipitate of tobacco pectins was obtained. The amount of precipitate in the graduated cylinder was observed. Each of these precipitates of tobacco pectins was suitable for use in the preparation of a reconstituted tobacco sheet in accordance with the methods taught by the present invention. The results obtained in these experiments are presented in Table IV below:

TABLE IV

Reagent:	Volume of coagulated tobacco pectins
Pentasodium tripolyphosphate	15
Sodium hexametaphosphate	5
Diammonium monohydrogen orthophosphate (DAP)	15
Trisodium orthophosphate	15
Disodium monohydrogen orthophosphate	15
Dihydrogen monosodium orthophosphate	5
Versene (tetrasodium salt of ethylenediaminetetraacetic acid)	15

Example 11

One hundred grams of burley tobacco stems were covered with distilled water, leached for 30 minutes and drained. A solution of 10 grams of Versene (tetrasodium salt of ethylenediamine-tetraacetic acid) in 600 ml. of water was added to the stems and the mixture was made basic with aqueous sodium hydroxide. The resulting mixture was then heated for 1 hour at room temperature. The entire mixture (a pulp) was homogenized in a Waring Blender and the solids content was determined to be 5% by weight. A 200 gram portion of the pulp, thus prepared, was combined with 2 grams of glycerin, as a humectant, and then cast into a film of 50 mils thickness by the use of a Gardiner casting knife. The resultant sheet was found to have satisfactory physical properties and, upon burning, exhibited a very pleasing aroma.

Example 12

Green bright tobacco leaves were soaked in isopropanol to remove sugars and chlorophyll. The midrib was stripped out of the leaves leaving the web. One thousand grams of the web (82 grams on a solids basis) were placed in

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a porcelain bucket and covered with boiling distilled water containing 9.3 grams of diammonium monohydrogen orthophosphate (DAP). The DAP was added on the basis of 10 grams of DAP per 100 grams of stems (which contained 12% moisture, by weight). The resulting mixture was boiled at low heat for one hour. The juice was then first expressed from the mixture by hand. Solids, for example cellulose and sand, were then further separated from the juice by centrifuging. The clear juice from both operations was then mixed with 70% ethanol to form a gel, which was then squeezed out in cheese cloth. The gel was transferred to a Buchner funnel where it was washed first with acetone and then with ethyl ether. Finally, the gel was placed in a vacuum desiccator and dried. The gel was suitable for use in the preparation of a reconstituted tobacco in accordance with the teachings of the present invention.

Example 13

Burley tobacco stalks were separated into cortical tissue, woody tissue, and pith. A sample of each was steam cooked with 10% sodium carbonate for 30 minutes at atmospheric pressure and for 20 minutes at 20 p.s.i.g. After this cooking, the woody tissue was still hard and could not be pulped in a Waring Blendor. The cortical tissue and pith were soft and pulpable. Each of these latter two preparations were suitable for use in the preparation of reconstituted tobacco.

Example 14

Coarse ground bright stem tobacco fines (30 grams) were washed thoroughly in cold water, and then placed in a boiling aqueous solution of 3 grams of diammonium monohydrogen orthophosphate and cooked for 5 minutes. The mixture was then placed in a Waring Blendor. After a very short period in the blendor, the mixture was converted to a viscous, fine, impalpable slurry; wherein the tobacco stem particles had been completely separated to units of cellular size. This impalpable mass was suitable for use as a binder in reconstituted tobacco. When remixed with some of the cold water washings removed in the first step, the impalpable mass immediately became viscous and eventually jelled to a soft mass.

For further comparison, a similar sample of coarse ground bright stem fines (30 grams), was dispersed in boiling water and cooked for about 30 minutes with three grams of diammonium monohydrogen orthophosphate. The pH of this mixture was brought to a value of 7.1 by the addition to the mixture of 30% aqueous ammonia. The granules of tobacco could then be reduced to a palpable pulp, similar to the soft mass produced in the experiment described in the first part of this example.

Example 15

Burley tobacco stems (½ pound) were covered with distilled water, allowed to stand several hours and the water decanted. This step was repeated several times and finally the stems were covered with distilled water containing 30 ml. of concentrated HCl and left overnight at 24° C.

After standing overnight the acidic water was decanted and the stems washed repeatedly free of HCl until the wash water gave no precipitate when treated with silver nitrate solution. The stems were then covered with distilled water containing 15 grams of sodium carbonate and left overnight at 24° C. The pH of the sample, the following morning, was 8.8. The stems were swollen and soft and were easily disintegrated with the fingers and the mixture could be homogenized and employed, as a binder in reconstituted tobacco.

Example 16

The apparatus employed in this experiment was large scale equipment, comprising a 200 gallon conical bottom, open top, stainless steel tank, fitted with a Cowles high speed mixer.

One hundred and forty-eight gallons of water were placed in the tank and heated to a temperature of 200° F. One hundred and twenty-eight pounds of bright tobacco (milled to pass a 6 mesh per inch sieve) stems were added, while operating the Cowles mixer at a low speed. Almost immediately after the addition of the tobacco, nine and one-quarter pounds of diammonium monohydrogen orthophosphate (technical grade) were added to the mixture. Ammonia (assaying 28%, by weight NH_3) was added to adjust the pH to 7.1. The mixing speed was increased to 1700 r.p.m. After a period of 3 minutes the temperature of the mixture was 194° F. Most of the particles in the mixture were soft enough to be smeared by hand and the mixture had a jelly-like consistency. The mixing was continued for 1 hour to obtain the highest possible state of disintegration, although a 15 minute period appeared to be sufficient for this purpose.

The viscosity of the mixture was found to be 10,400 cps. and its solids content was found to be 8.05% by weight.

This mixture was then employed as a binder, being pumped through a filter to storage tanks and subsequently sprayed on tobacco by the method and equipment described in U.S. Patent 2,734,513. It was applied at the rate of 3 grams per square foot to form a reconstituted tobacco sheet having the following physical properties:

Basis weight, gms./ft.² (1)	10.2
Moisture content (percent by weight)	13.0
Tensile, kg./in.	0.82
Fold tensile, kg./in.	0.82
Work-to-break, gm. cm./ft.² (2)	16.0

(1) Weight of all the ingredients, including tobacco pulp, conditioned free of moisture.
(2) Measured by an Integrator attached to the Instron Tensile Tester.

Example 17

Burley tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then dried and ground and used to make a binder as follows:

One hundred parts of water were brought to a temperature of 195° C., and to this were added:

7.00 parts by weight (dry basis) of the washed burley stems,
1.05 parts diammonium phosphate, and
0.70 part of glycerin, as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 7.1 but no higher than 9.0.

The mixture was then stirred for one hour and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 18

Burley tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then used directly in the wet condition to make a binder as follows:

One hundred parts of water were brought to a temperature of 195° C., and to this were added:

7.00 parts by weight (dry basis) of the washed burley stems,
1.05 parts diammonium phosphate, and
0.70 part of a glycerin, as a humectant.

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Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 7.1 but no higher than 9.0.

The mixture was then stirred for one hour and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 19

Burley tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then dried and ground and used to make a binder as follows:

One hundred parts of water were brought to a temperature of 195° C., and to this were added:

7.00 parts by weight (dry basis) of the washed burley stems,
1.05 parts diammonium phosphate, and
0.70 part of triethylene glycol (TEG), as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 7.1 but no higher than 9.0.

The mixture was then stirred for one hour and subsequently refined in a disc type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plants parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 20

Burley tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then used directly in the wet condition to make a binder as follows:

One hundred parts of water were brought to a temperature of 195° C., and to this were added:

7.00 parts by weight (dry basis) of the washed burley stems,
1.05 parts diammonium phosphate, and
0.70 part of triethylene glycol (TEG), as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 7.1 but no higher than 9.0.

The mixture was then stirred for one hour and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 21

Bright tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then dried and ground and used to make a binder as follows:

One hundred parts of water were brought to a temperature of 100° C., and to this were added:

7.00 parts by weight (dry basis) of the washed bright (flue cured) stems,
1.05 parts diammonium phosphate, and
0.70 part of glycerin, as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 8.0 but no higher than 8.5.

The mixture was then stirred for four hours and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 22

Bright tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then used directly in the wet condition to make a binder as follows:

One hundred parts of water were brought to a temperature of 100° C., and to this were added:

7.00 parts by weight (dry basis) of the washed bright (flue cured) stems,
1.05 parts diammonium phosphate, and
0.70 part of glycerin, as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 8.0 but no higher than 8.5.

The mixture was then stirred for four hours and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 23

Bright tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then dried and ground and used to make a binder as follows:

One hundred parts of water were brought to a temperature of 100° C., and to this were added:

7.00 parts by weight (dry basis) of the washed bright (flue cured) stems,
1.05 parts diammonium phosphate, and
0.70 part of triethylene glycol (TEG), as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 8.0 but no higher than 8.5.

The mixture was then stirred for four hours and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 24

Bright tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then used directly in the wet condition to make a binder as follows:

One hundred parts of water were brought to a temperature of 100° C., and to this were added:

7.00 parts by weight (dry basis) of the washed bright (flue cured) stems,
1.05 parts diammonium phosphate, and
0.70 part of triethylene glycol (TEG), as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 8.0 but no higher than 8.5.

The mixture was then stirred for four hours and subsequently refined in a disk type refiner until better than

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99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 33

In the acid wash treatment of tobacco parts for removal of alkaline earth minerals of the tobacco pectin, it is necessary to use quite large volumes of water if it is desired to reduce the soluble anion content of the product to a very low level. In this case, the use of a cation exchange resin in a closed loop system with the tobacco parts can make it possible to conduct the process with limited amounts of water and acid.

The use of a resin (such as Dowex 50W) in this way does not at all alter the principles of this form of treatment since the resin merely serves as a convenient reservoir of acidity, continuously reconditioning effluent from the stems for reuse in the extraction.

In the following example, the use of a cation exchange resin permits the treatment of stems with a small fraction of their natural nitrate ion content. Hydrochloric acid is used at intervals to regenerate the resin, but never directly contacts the tobacco.

The apparatus used for ion-exchange extraction of calcium from tobacco stems consisted of a 28 inch diameter washing column fitted with a 20 mesh screen to support the charge of stems and a 6 inch diameter Pyrex column filled with 13 pounds of Dowex 50W-8 acid-form ion exchange resin beads. The valves and piping permitted a pump to be used either for recycling waters from the stem charge through the column or for separately regenerating the resin with hydrochloric acid solution.

In operation, fifty pounds of stems were placed in the washing column and rinsed with cold water until they were nearly free of water-soluble material. Then, the effluent waters were diverted through the ion exchange bed, returning by gravity to the top of stem charge. When the aqueous extract from one pound of burley stems was added to the recycling waters, a pH of between 1.5 and 2.0 was soon attained. During the treatment, the waters flowing from the resin column were monitored for their calcium content. (An appreciable precipitate from 30 ml. of resin effluent treated with ammonium oxalate and made basic with ammonia indicated that the resin was saturated with calcium and needed to be regenerated with hydrochloric acid.) After sixteen hours of operation, and one regeneration of the ion exchange resin, the treatment was considered complete. A subsequent analysis showed that the calcium content of the stems had been reduced to less than one-sixth of the starting value of 4.5% CaO. The stems were rinsed twice with cold distilled water, pressed lightly, and redried in a tobacco processing oven to a final moisture content of about 4%, and ground to pass a 40 mesh screen. When a small sample of this powder was stirred with a little cold ammonia, it dispersed immediately, forming a stiff paste.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 34

Twenty grams of tobacco fines were washed in 1 liter of distilled water to get rid of the tobacco solubles. The fines were then admixed with 1 N hydrochloric acid until the mixture had a pH of 1.35. The pH was checked on a Beckman pH machine and the curves were also plotted against a titration of distilled water.

After titration, the acid was washed off the fines with three 400 ml. portions of distilled water. The fines were redistributed in 400 ml. of distilled water and sodium bicarbonate was added to bring the pH to 7. The slurry was cast at a thickness of 30–50 mils and dried to form a sheet and the resulting sheet was used as the adhesive binder in sandwich type reconstituted tobacco sheet.

Example 35

Twenty grams of tobacco fines were washed in 1 liter of distilled water to get rid of the tobacco solubles. The fines were then admixed with 1 N hydrochloric acid until the mixture had a pH of 1.35. The pH was checked on a Beckman pH machine, and the curves were also plotted against a titration of distilled water.

After titration, the acid was washed off the fines with a 400 ml. portion of distilled water. The fines were redistributed in 400 ml. of distilled water and sodium bicarbonate was added to bring the pH to 7. The slurry thickened to a viscous mass, which is evidence that the pectin had been solubilized. The slurry was cast at a thickness of 30–50 mils and dried to form a sheet and the resulting sheet was used as the adhesive binder in sandwich type reconstituted tobacco sheet.

Examples 28–30

In each of these three examples, 20 gms. of tobacco fines were washed in 1 liter of distilled water to get rid of the tobacco solubles. The fines were then admixed with 1 N hydrochloric acid until the mixture had a pH of 1.35. The pH was checked on a Beckman pH machine and the curves were also plotted against the titration of distilled water.

After titration, the acid was washed off the fines with three 100 ml. portions of distilled water. The fines were redistributed in 400 ml. of distilled water and a neutralization agent, as indicated in Table V, was added to bring the pH to the value shown in Table V. The slurry in each case thickened to a viscous mass which is evidence that the pectin had been solubilized. The slurry was cast at a thickness of 30–50 mils and dried to form sheets. The binders formed in this way were tested to determine their physical properties. These properties are shown in Table V below. The binder was used as the adhesive in a sandwich type reconstituted tobacco sheet.

TABLE V.—PROPERTIES OF BINDER FILM MADE BY ACID WASH PROCESS

Example	1	28	29	30
pH	7.1	7.2	7.2	7.1
Basis Wt. gm./ft.²	4.34	4.79	4.73	4.73
Mg. absorbed per sample	(2)	(2)	(2)	(2)
Integrator Count	844	848	848	848
Percent Moisture	11.3	11.6	12.1	12.1
Percent Elongation	1.88	1.10	1.88	1.88
Tensile Pli	2.37	2.33	2.38	2.38
Tensile Coefficient, kg. per gm. of basis wt.	6.42	6.33	6.38	6.38
Work Coefficient, gm. cm./sq. in.	4.77	4.58	4.82	4.82

(1) Conc. NH4OH.
(2) 1% KOH.
(3) 1% NaOH.

In the table the terms have the following meanings:

(1) Integrator count is proportional to the area under the stress-strain curve recorded by an Instron Test Machine.

(2) Percent moisture=The percentage of moisture contained in the entire sheet (wet basis).

(3) Percent elongation=The percentage elongation, at breakage, on an Instron Test Machine.

(4) Tensile kg./in.=The breaking strength of a 10 cm. test strip which is 1 inch wide; an average of 10 strips.

(5) Tensile coefficient=kg. per gm. of basis weight.

(6) Work coefficient gm./sq. in.=Work coefficient is proportional to the product of the tensile strength and the elongation.

Example 31

The following ingredients were mixed into 900 parts of hot water and heated for 3 hours at 85°–90° C., main-

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BEST IMAGE

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raising a pH of 7.0 by adding small amounts of aqueous ammonia.

	Parts
Mixed manufacturing byproducts and scrap tobacco	87.9
Diammonium orthophosphate (DAP)	5.0
Glycerin	1.0
Corn syrup	1.0
Potassium sorbate	0.14

The slurry was then refined in a one gallon capacity Waring Blendor for 5 minutes. With this amount of refining, the fibrous portion of the composition was seen to be well dispersed and thoroughly interlocked when examined in a droplet at a 1:9 dilution. This examination was facilitated by the addition of a small amount of Congo Red dye.

The composition was cast on stainless steel panels with an eight inch angle knife set for wet film thicknesses of 0.020 to 0.045 inch, and dried on a steam table.

The wet strength of the films was determined on a Scott Serigraph IP-2 using 1 inch width strips. The center of each strip was wet with distilled water on both sides and allowed to become thoroughly wet through (30 seconds or one minute) before actuating the tensile mechanism. These films showed the extraordinary high wet film strength of 140 grams at a film weight of 9.5 grams per square foot. A high wet strength is highly desirable in the preparation of cigarette filter, and assures that the subsequent manufacturing performance of this material will exceed that of the finest grades of leaf tobacco. Such strength is uniquely obtainable in the presence of the liberated form of pectin described in this disclosure.

We claim:

Tobacco sheet material comprising finely divided tobacco bonded together by an adhesive derived from tobacco plant parts containing pectins having alkaline earth metal cross-linkages, said adhesive being prepared by the process comprising the steps of:

(1) Contacting said tobacco plant parts with a treating solution containing a reagent comprising diammonium acid phosphate to destroy the alkaline earth metal cross-linkages.

(2) At least partially releasing the resulting tobacco pectins from the interstices of the treated tobacco plant parts.

(3) Depositing said tobacco pectins on the treated plant parts, and

(4) Recovering the resulting mixture.

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SAMUEL KOREN, *Primary Examiner.*

MELVIN D. REIN, *Examiner.*

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PHILIP MORRIS U. S. A.

INTER-OFFICE CORRESPONDENCE

RICHMOND, VIRGINIA

PERSONAL & CONFIDENTIAL

To: Mr. C. H. Goldsmith Date: November 30, 1977

From: R. B. Seligman

Subject: Council for Tobacco Research Program (CTR)

On November 22, 1977, Tom Osdene represented Philip Morris at a CTR program review session. In addition to his trip report, Tom wrote a memo to me commenting about what he heard. A copy of this personal and confidential memo is attached.

As you will see when you read Tom's memo, he was very disturbed with the trends that are developing in the CTR program. I thought I should bring this to your attention because you are a member of an executive committee of the CTR. Also, because of the nature of Tom's memo, I have given it very limited distribution.

If we can provide more information, please let me know.

RBS/sgr

Attachment (1)

cc: Mr. W. W. McDowell (with attachment)

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restricted film alone

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PHILIP MORRIS U. S. A.

INTER-OFFICE CORRESPONDENCE

RICHMOND, VIRGINIA

CONFIDENTIAL

To: Dr. R. B. Seligman Date: November 29, 1977

From: T. S. Osdene

Subject: Some Comments about the CTR Program

I was amated at the trend that the CTR work is taking. For openers Dr. Donald H. Ford, a new staff member, makes the following quotes:

"Opiates and nicotine may be similar in action."

"We accept the fact that nicotine is habituating"

"There is a relationship between nicotine and the opiates."

Dr. Lee Abood, whose presentation showed a high degree of competence, has one of his aims a specific antagonist to nicotine. This is done by various chemical manipulations of nicotine and could well lead to a clinically acceptable antagonist.

Dr. Kreisher's work in the area of AAH, from my point of view, stands out with the proposition that smoking causes lung cancer. The rest of the work seems to justify this approach. I am very surprised about the extensive human clinical data which is now being sought and feel that after four years of this project nobody has the slightest idea where it is going, where it is, or what it is trying to prove.

It is my strong feeling that with the progress that has been claimed, we are in the process of digging our own grave. I believe that the program as set up has the potential of great damage to the industry and I strongly urge that the whole relationship of our Company to CTR be carefully reviewed. I am very much afraid that the direction of the work being taken by CTR is totally detrimental to our position and undermines the public posture we have taken to outsiders.

TSO/nro

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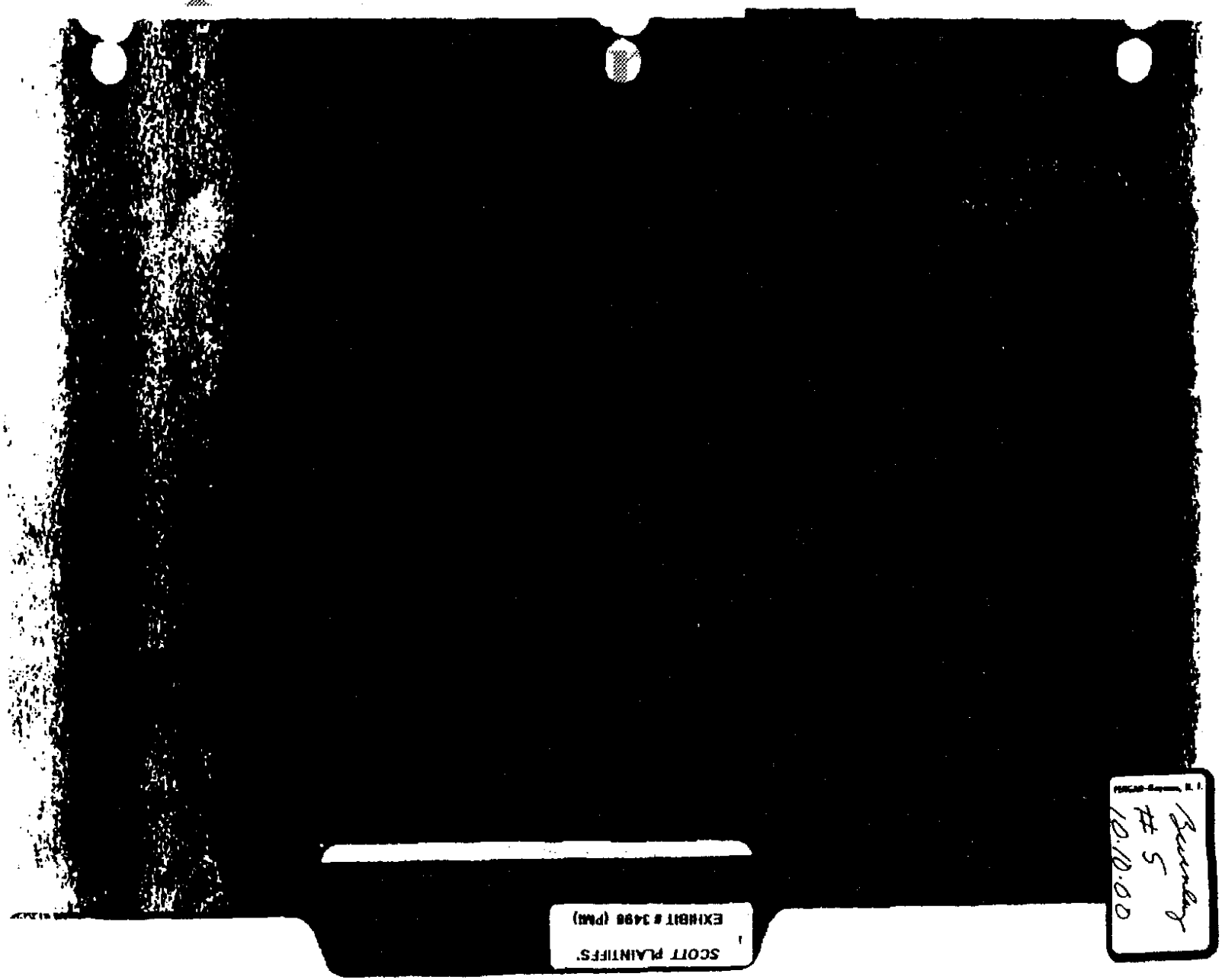

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PHILIP MORRIS

U.S.A.

RESEARCH CENTER: P.O. BOX 26583, RICHMOND, VIRGINIA 23261 TELEPHONE (804) 271-2000

March 15, 1978

78-00185

Mr. Jack Hind
630 Yucca Road
Naples, FL 33940

Dear Jack:

Hi, good friend. When Bob brought me your letter I was so very pleased to have word from you, even at the expense of being given the chore of rebutting the accompanying newspaper editorial.

What do you want rebutted? The idea that habitual smokers are under pressure? I can't rebut that. That nonsmokers have to breathe air contaminated by someone else? That doesn't even merit rebutting; everybody breathes air contaminated by someone else, even when no cigarettes are being smoked.

I suspect you'd like me to comment upon the 5% physical addiction/95% psychological dependence conceptualization proposed by the American Cancer Society.

I'm puzzled as to the source of the percentages. I've not seen the 5%/95% ratio anywhere in the literature, not even as a speculative judgment. I cannot conceive of any hard data to support such values. Such statements strike me as being grist for the P.R. mill, invented casually by someone naive about the ways of science, and indifferent about the rigors of science.

We cannot so cavalierly dismiss the more general notion of the two components of the smoking habit, i.e. the nicotine reinforcement and the psychological/social reinforcement. In fact, we, too, are thinking in such terms.

As for the nicotine reinforcement, we are beginning to suspect that rather than there being an attempt to maintain some blood constancy level, the smoker is seeking the spike effects of blood nicotine which occur immediately upon inhalation. These effects may well be so rewarding that the habitual smoker overloads himself in seeking them, with high blood nicotine levels occurring after the fact, and not essential to the reinforcement process.

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The psychological/social reinforcement is the result of complex learning processes, in our view. Certain situations elicit smoking for the effects of nicotine. By association, cues in those situations come to have smoking-eliciting power, such that even in the absence of need for nicotine the cues can trigger the smoking sequence. Thus the smoker smokes much of the time not for nicotine but because the act has been set in motion by events in his life-space. This kind of smoking, however, is contingent upon the associative learning which in turn has occurred because of the rewarding effects of smoke inhalation.

There is yet another kind of psychological/sociological smoking in which nicotine rewards have not been present. This is to be seen in the noninhaler for whom smoking enhances his social image; a strong determinant in the initiation of the smoking habit among the young.

So we do subscribe to a two-factor smoking theory as attributed to the American Cancer Society by the writer of the article you sent us. We do not, however, endorse the 5%/95% ratio. That's poppy-cock.

Bob showed me the picture of your sailing rig. Was that you in it? Lucky dog.

Best regards,

Bill

Bill Dunn

jh

cc: Dr. R. B. Seligman

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SMOKE IMPACT FROM A PSYCHOLOGIST'S VANTAGE POINT

T. R. Schori
Behavioral Research Laboratory
Philip Morris, USA

October, 1974

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BACKGROUND

It is immediately apparent to anyone that has ever tried 100% burley cigarettes and 100% bright cigarettes that the two tobacco types result in very different sensory responses. These differences are primarily in smoke (or inhalation impact)--the smoke impact resulting from the burley tobacco being considerably higher than that from the bright tobacco. Smoke impact is a convenient term that has been applied to the individual's degree of awareness of the presence of smoke in the back of the throat (a slight variation of Dunn's definition, c.f., Schori, 1974a), and is probably analogous to what some smokers may describe as smoke streagth.

Sensory Model

Previously (Schori, 1974b), I proposed a sensory model of smoke impact, that is, of the sensory experiences that are termed smoke impact. This model is shown below:

smoke ⟶ receptor ⟶ smoke impact

From this model, it can be seen that smoke impinges on certain receptors. The activation of these receptors results in certain sensory experiences which we call smoke impact. The degree of impact (or more accurately, the degree of one's awareness of the presence of smoke in the back of the throat) is determined by the number of receptors activated and/or the frequency of each receptor's activation. Located in the back of the throat, the receptors, themselves, are in all likelihood pain receptors. The receptors provide the individual with information concerning the nature of the smoke he is inhaling and, accordingly, allow him to voluntarily terminate inhalation before circumstances are such that respiratory tract aversive consequences might result.

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Smoke Constituent Responsible for Impact

Specifically, though, what component in cigarette smoke is responsible for activating these receptors and, thus, for giving rise to the sensory experiences that we call smoke impact? At the time that the sensory model for smoke impact was first conceptualized (Schori, 1974b), I was not prepared to hypothesize as to which specific component of cigarette smoke causes smoke impact. However, I am now prepared to hypothesize. It is nicotine. Other smoke constituents may also stimulate these receptors (remember pain receptors tend to respond to various forms of stimulation) but nicotine is the main constituent in smoke to do so and, thus, smoke impact is primarily determined by the nicotine in the smoke.

Suggesting that smoke impact is due to nicotine, however, is nothing new. The important factor, though, is not the amount of nicotine in the smoke per se but rather it is the amount of free nicotine in the smoke, which determines degree of smoke impact. This appears to have been recognized long ago by Shmuk (1939) who utilized the "nicotine number," which equals % total nicotine/% free nicotine, as an objective indicator of the strength of tobacco smoke. Thus, when total nicotine is held constant, the strength of the smoke will increase with decreases in the nicotine number--which is exactly the same thing as saying that strength (or smoke impact) will increase with increases in the amount of free nicotine in the smoke. More recently, Shakhnovskii (1959) also observed that smoke strength is related to the amount of free nicotine in the smoke. Some Russian investigators (Debrand, 1974) have reported smoke strength to be logarithmically related to nicotine in the smoke; this is counter to the current view that sensation is exponentially related to stimulus intensity (Stevens, 1961).

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This discussion may be confusing unless it is apparent what I mean by total nicotine and free nicotine. The distinction between the two was initially even somewhat confusing to the analytic chemists involved in making nicotine determinations in smoke; their first response to my query was that virtually 100% of the nicotine present in smoke is free nicotine. This response, however, was erroneous. Not all the nicotine present in tobacco smoke is free nicotine.

Importance of pH

The way in which nicotine is typically reported can be misleading. This is due to the manner in which nicotine determinations are made. For instance, cigarettes X and Y may both be reported to deliver (based upon the standard smoking machine test) 2 mg nicotine/cigt. However, a given smoker may actually inhale much more free nicotine from cigarette X than from cigarette Y. Likewise, cigarette W may deliver 1 mg nicotine/cigt. while cigarette Z delivers 2 mg nicotine/cigt. Yet a given smoker may inhale equal amounts of free nicotine from cigarettes W and Z. This paradox results from the fact that in making the nicotine delivery determinations strong bases are employed to free or release the nicotine from its bonds with other elements (e.g., to free the nicotine from nicotine citrate). By using these strong bases, virtually all the nicotine is liberated. Nicotine delivery, then, as reported in analytical reports actually represents the total possible (or virtually so) free nicotine. It does not, though, represent the free nicotine that is actually available to the smoker--since the smoker is unable to employ strong bases in order to liberate all the nicotine, that is, at least not in his mouth--which is the important thing from a smoke impact point of view. Thus, the amount of free nicotine available to the smoker is determined by the degree of alkalinity (or pH) of the smoke as well as own degree of alkalinity. Therefore, the free nicotine inhaled by him is always some proportion of the total possible free nicotine.

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For our purposes, the amount of free nicotine available to the smoker is determined by the pH of the smoke, since we have no control over the pH of the smoker's mouth. As pH increases, there is a corresponding increase in the proportion (of the total possible free nicotine) that is actually liberated (Handy, 1971). This relationship is depicted in Figure 1. From this figure, it can be seen that as pH increases from 5½ to 9 there is a very sharp increase in the proportion of nicotine that is freed. Thus, it should be possible to increase smoke impact by artificially increasing pH.

Importance of Tobacco Constituents

Shmuk (1939) also discussed the usefulness, as an indicator of both taste quality and strength of tobacco smoke, of the ratio of carbohydrates to proteins (% carbohydrates/% proteins) in the tobacco. This ratio is generally referred to as Shmuk's number (Abdallah, 1970). According to Shmuk, as the Shmuk number increases, there will be corresponding decreases in smoke strength. In other words, when carbohydrates in the tobacco become relatively more plentiful than proteins, smoke strength will decrease; the reverse will result in increases in smoke strength. This suggests that we could increase smoke impact by the addition of proteins to the tobacco.

Why should the ratio of carbohydrates to proteins have any bearing on taste strength or smoke impact? The answer is simple. In combustion of tobacco, carbohydrates tend to form acids while proteins tend to form bases. Thus, the tendency is for the carbohydrates to reduce pH while proteins tend to increase pH. That is, carbohydrates and proteins in tobacco act as antagonists--with their ratio partially determining the total pH of the smoke. Therefore, as Shmuk's number decreases, there will be corresponding decreases in the proportion of free nicotine in the smoke and, accordingly, less smoke strength. One of the important carbohydrates

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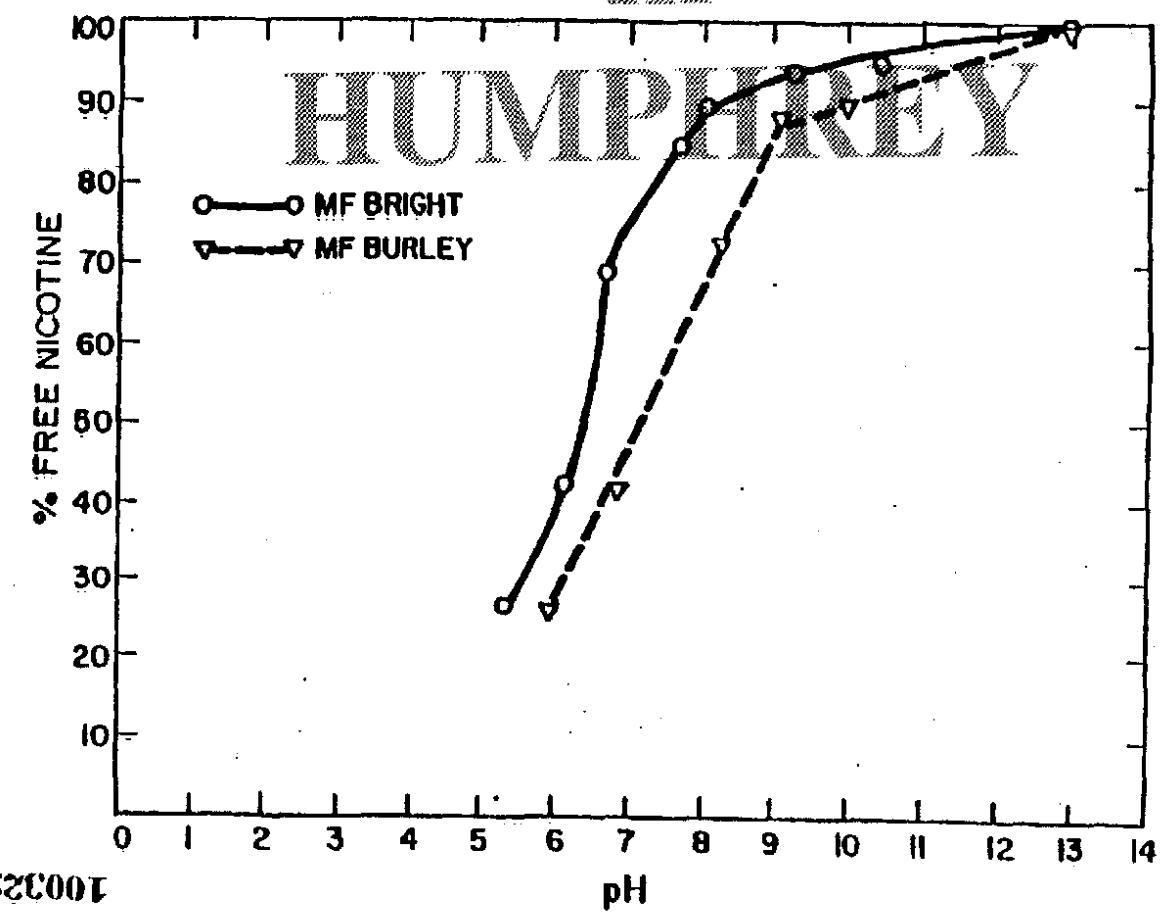

Figure 1. Percent free nicotine as a function of pH. Adapted from Handy, 1971.

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in tobacco is sugar (Elson, et al., 1972). The relationship between sugars in the tobacco and pH of the resulting smoke is shown in Figure 2. It is readily apparent from inspection of this figure that low concentrations of sugars are associated with high pH of the smoke and, thus, a proportionately large amount of free nicotine.

Shmuk's number (carbohydrates/proteins ratio) leaves something to be desired. It does not take into consideration the fact that numerous other constituents in tobacco in addition to proteins, e.g., ammonia, nitrates, etc., also tend to form bases upon combustion and, thus, tend to increase the total alkalinity (pH) of the tobacco smoke. Therefore, it would be more appropriate to use the ratio of carbohydrates to total nitrogenous substances, as an indicator of smoke strength or of smoke impact, i.e., 10x (% nitrogenous substances/% carbohydrates) which will be referred to as the N/C number. Since our primary interest is in increasing smoke strength or smoke impact, I have purposely inverted the ratio; it was multiplied by 10 so that numbers greater than 1 would normally be achieved. As the N/C number increases, there are corresponding increases in pH and, therefore, in free nicotine in the smoke. Thus large values of the N/C number should be associated with high strength and smoke impact while small values should be associated with low strength and smoke impact.

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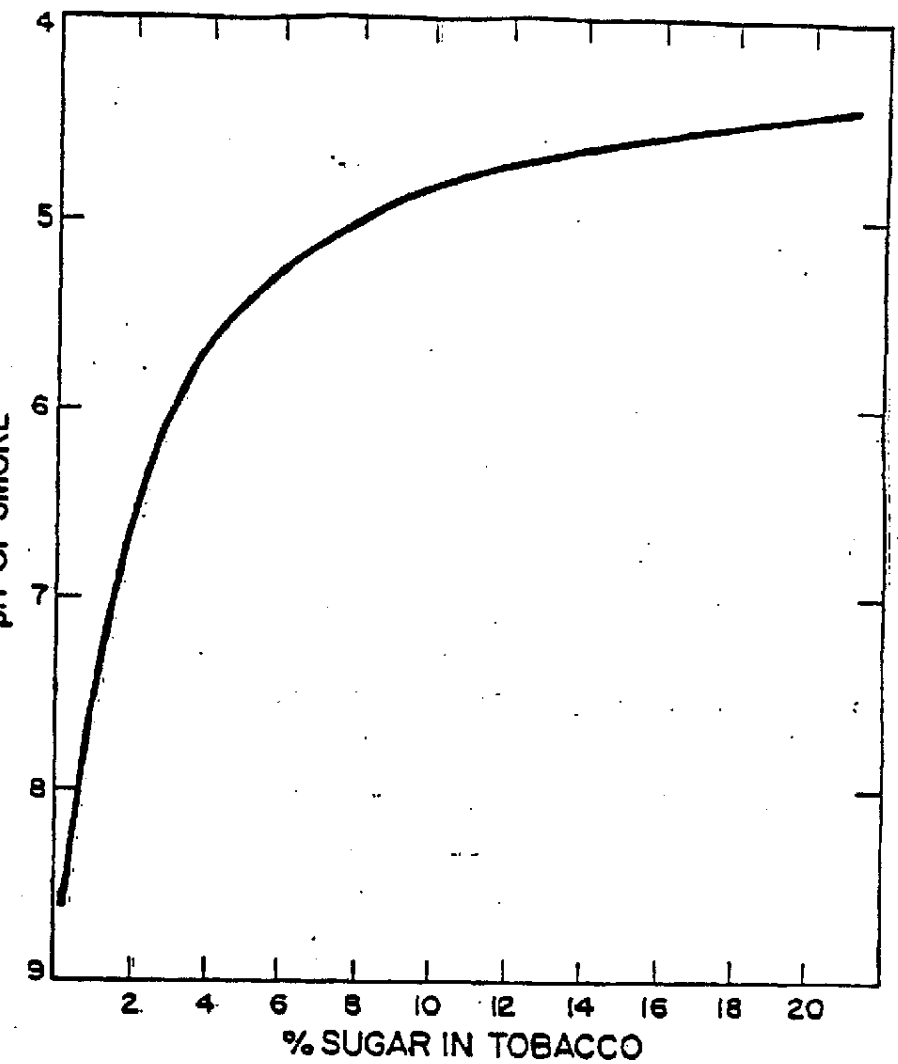

Figure 2. Smoke pH as a function of percent sugar concentration in tobacco. Adapted from Elson, et al., 1972.

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DISCUSSION

If my assumptions are correct, then, smoke impact is due to the amount of free nicotine in the smoke. The amount of free nicotine in the smoke is determined by two factors: (1) the total potential free nicotine in the smoke (which in our usual way of viewing it, is simply nicotine delivery in mg/cigt.); and (2) pH of the smoke, which determines the proportion of the nicotine that is actually liberated in the smoke.

Obviously, then, we should be able to increase smoke impact by increasing the total free nicotine potential in the smoke. When we have done this in various studies, e.g., Schori, 1971; Schori & Martin, 1974a, strength ratings (which are probably analogous to smoke impact ratings) have been found to increase. In these studies, the nicotine in the tobaccos was artificially augmented with nicotine citrate or nicotine tartrate, which itself influences the pH of the smoke slightly.

Also, we should be able to increase smoke impact by simply increasing the pH of the smoke and, thereby, causing more nicotine to be available to the smoker as free nicotine. We could also reduce impact by decreasing pH, however, this paper is primarily concerned with techniques for increasing smoke impact. I might mention, though, that Hind (1974) is currently investigating pH reduction methods, similar to those of Shmuk, et al. (1932), viz., by the addition of acids to the tobaccos. It is apparent that we could add nitrogenous substances to the tobacco in order to increase the pH. However, such techniques (e.g., the addition of ammonia) have not proved too successful since they tend also to produce unpleasant side effects, such as making the side stream smoke very noxious. But let us consider readily available means of increasing pH in tobacco smoke, that is, those that can be accomplished by varying our existing blends. In Table 1, a breakdown of the various

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Table 1*

Constituents of Major Tobacco Types

	Bright	Burley	Maryland	Turkish
Nitrogenous Constituents				
Total Nitrogen	(1.61)	(3.26)	(2.30)	(2.17)
Volatile Bases (VB) as Ammonia	0.28	0.07	0.37	0.39
Nicotine	1.93	2.91	1.27	1.61
% Volatile Bases minus Nicotine as Ammonia	0.08	0.32	0.23	0.16
% Ammonia	0.02	0.16	0.13	0.10
% alpha-Amino Nitrogen	0.05	0.17	0.06	0.10
% Glutamine	0.20	0.36	0.35	0.17
% Asparagine	0.15	0.08	0.32	0.46
% Glutamic Acid	0.04	0.12	0.11	0.09
% Aspartic Acid	0.02	0.14	0.04	0.23
% Nitrate as NO_3	Trace	1.76	0.09	0.23
% Protein	1.69	11.06	10.86	7.44
Total	8.58	16.16	12.83	10.10
Carbohydrate Constituents				
% Reducing Sugars (Before Inversion) as Dextrose	22.88	Trace	Trace	12.39
% Pectin as Calcium Pectate	4.18	9.91	12.11	6.77
% Crude Fiber	7.68	9.26	21.79	6.63
% Lignin	6.37	5.62	10.26	6.43
Total	41.83	24.86	44.19	32.22
Acids				
pH	5.48	5.84	6.60	6.90
% Volatile Acids (VA) as Acetic	0.15	0.16	0.09	0.19
% Formic Acid	0.04	0.03	0.02	0.08
% Volatile Acids (VA) minus Formic as Acetic	0.08	0.07	0.06	0.09
% Malic Acid	1.01	6.76	2.15	3.07
% Citric Acid	0.78	0.22	2.90	1.01
% Oxalic Acid	0.01	3.04	2.70	3.16
Aromatics				
% Volatile Oil	0.18	0.14	0.14	2.25
% Alcohol-Soluble Resins	9.08	9.27	8.94	11.28
Mineral Constituents				
% Ash	18.01	24.63	21.90	14.76
% Calcium as CaO	2.22	8.01	4.39	4.22
% Potassium as K_2O	2.47	6.22	4.48	2.33
% Magnesium as MgO	0.26	1.29	1.03	0.69
% Chlorine as Cl	0.84	0.71	0.16	0.69
% Phosphorus as P_2O_5	0.21	0.17	0.13	0.47
% Sulfur as SO_4	1.23	1.38	3.14	1.86
Summary				
Nicotine	1.93	2.91	1.27	1.61
pH	5.45	5.84	6.56	6.90
Shmuk's Number	7.18	3.26	1.38	4.31
N/C Number	1.06	7.46	6.81	3.13

*Adapted from Burton & Hawley, 1956; Hyader & Hoffmann, 1967.

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constituents in the major tobacco types is shown (c.f. Harlan & Moseley, 1955; and partially c.f. Wynder & Hoffmann, 1967). Whether or not all the constituents in tobaccos are accurately reflected in this table is questionable. For instance, the relative concentrations of malic, citric, and oxalic acid differ slightly from those reported by Harvey, et al. (1970). However, the values shown in the table at least grossly represent the relative concentrations of the various constituents in tobacco.

Tobacco Types

The major tobaccos in our blends are burley and bright. Therefore, the values shown in Table 1 for these tobaccos are of primary interest. It will be noticed that total nicotine is higher in burley than in bright. Thus, there is a potential for greater amounts of free nicotine in the smoke from burley tobacco than there is from bright tobacco. In addition, the pH of the burley is higher than that of the bright. And this difference in pH is actually magnified in the smoke, since pH of smoke is considerably higher than that of the tobacco from which it was generated (due to the products of combustion). Because of the higher pH, proportionately more of the nicotine is present in the free state in burley smoke than in bright smoke. These two factors in combination, then, are responsible for the much higher impact in 100% burley cigarettes than in 100% bright cigarettes, i.e., both the higher total nicotine and the high pH that are found in burley tobacco. Even if some particular burley and bright tobaccos had equal amounts of total nicotine, the burley tobacco would have more free nicotine in its smoke as a consequence of its higher pH and, therefore, its smoke impact would still be higher than that of the bright. Likewise, even if some particular burley and bright tobaccos had equal pH, the burley tobacco would have more free nicotine in its smoke as a result of its greater amount of total nicotine. Accordingly, its impact would still be higher than that of the bright.

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Since it is a given that a 100% burley cigarette results in greater smoke impact than a 100% bright cigarette, Shmuk's number (carbohydrates/proteins) should be lower for burley tobacco than for bright tobacco. Inspection of Table 1 reveals that burley does result in a lower Shmuk number than bright tobacco. Likewise, the N/C number (nitrogenous substances/carbohydrates) should be higher for burley than for bright. And from Table 1 it can be seen that the N/C number is higher for burley tobacco than for bright. Both Shmuk's number and the N/C number, then, should provide some predictive information concerning smoke impact. However, since the N/C number takes into consideration total nitrogenous substances rather than just proteins, its utilization may be more useful as an aid in predicting impact than Shmuk's number.

Taking into consideration these known differences between burley and bright tobaccos, we can with great ease increase smoke impact by increasing the proportion of burley that we put in the blend. By doing this, we can take advantage both of the higher nicotine concentration in burley as well as its higher concentration of other nitrogenous substances (besides nicotine), which are responsible for the higher pH in burley tobacco and, accordingly, for the higher proportion of free nicotine in the smoke.

In addition, we could reduce the carbohydrate concentration in the tobacco--eliminating components that upon combustion tend to form acids. Effectively, this would result in higher pH and therefore a higher proportion of free nicotine in the smoke. Thus, the elimination of carbohydrates would result in greater smoke impact. Easier than doing this, however, would be to eliminate some of the carbohydrates that we currently add to our tobaccos. With burley, we add carbohydrates, viz., sugars, as flavoring. Actually, we know from the preceding discussion that the addition of sugar will act to reduce pH and, thus, reduce the availability of free nicotine in the smoke. Therefore, the addition of sugar acts, in this fashion, to reduce smoke impact.

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HTV MMT

Applications

Are we, in any of our studies, taking advantage of what we suspect to be the relationships between smoke impact and free nicotine, pH, the relative concentrations of nitrogenous and carbohydrates constituents of tobacco? Yes, currently we have two such studies underway--both of which are concerned with smoke impact in low delivery cigarettes and involve manipulations of free nicotine. One of these studies (see proposal in Schori & Martin, 1974a) involves a relatively direct manipulation of free nicotine, i.e., varying amounts of nicotine salts will be added to the tobacco, in 10 mg tar cigarettes. In the other study (Schori, et al., 1974a & b), we will be making two types of relatively indirect manipulations of free nicotine in 5-6 mg tar cigarettes. Specifically, in the latter study, free nicotine will be varied both by varying the proportion of burley in the blend (the higher the burley concentration, the higher the total nicotine as well as the proportion of free nicotine) and by varying the carbohydrate concentration (the less the carbohydrates the greater the pH and, accordingly, the greater the proportion of free nicotine). Although it appears likely that free nicotine is primarily responsible for the smoker's sensation of smoke impact, psychological factors should not be overlooked. There is some evidence (Schori & Martin, 1974b) that filtration characteristics of the cigarette can be used, by us, to simulate greater impact than the amount of free nicotine would suggest, i.e., to fool the smoker. Taking this possibility into consideration, two types of filtration systems will be used in the Schori, et al. (1974a & b) study to achieve the 5-6 mg tar delivery levels.

Alternative Explanation of Impact

It has been suggested (Fagan, 1974) that pH (or the effective concentration of hydrogen ions) itself may be responsible for the sensation termed smoke impact. This suggestion has some merit. However, when we have augmented the natural nicotine in the tobacco (e.g., Schori, 1971) pH has remained nearly constant, as the total concentration of nicotine increased. And we have found smoke impact

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(actually smoke strength) to increase as total available nicotine increases. This suggests, then, that the influence of nicotine, i.e., free nicotine, is greater than that of pH on the resulting subjective impression of smoke impact. Actually, the addition of nicotine salts should, if anything, slightly decrease pH. Thus, if smoke impact was determined by pH per se, we would expect that corresponding to our addition of nicotine salts there would be decreases in smoke impact--which there were not.

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SUMMARY

Smoke impact is a convenient term that has been applied to the smoker's degree of awareness of the presence of tobacco smoke in the back of the throat. Smoke impact is probably analogous, for most smokers, to smoke strength. More than likely, the sensation is mediated by pain receptors in the back of the throat. But what constituent in the smoke activates these receptors? Pain receptors respond to various forms of stimulation, however, it is felt that the primary constituent of tobacco smoke which activates them is nicotine. However, it is not nicotine per se that is responsible but rather the amount of free nicotine. Thus, degree of smoke impact is viewed as being determined by the amount of free nicotine in the smoke.

The amount of free nicotine in the smoke depends upon: (1) total nicotine, and (2) pH of the smoke. The higher the pH, the greater the proportion of free nicotine and, accordingly, the greater the smoke impact. Also related to smoke impact is the N/C number, i.e., the ratio of % total nitrogenous substances/% carbohydrates. As the N/C number increases, there will probably be corresponding increases in smoke impact. The explanation for this is that the nitrogenous substances, upon the combustion of the tobacco, contribute to the alkalinity of the smoke while the carbohydrates contribute to its acidity. Thus their ratio influences the pH of the smoke, and therefore the proportion of nicotine that is liberated in the smoke.

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PHILIP MORRIS U. S. A.

INTER-OFFICE CORRESPONDENCE

RICHMOND, VIRGINIA

To: Dr. J. L. Charles Date: July 20, 1982

From: V. J. DeNoble

Subject: Project Number 1610 (Behavioral Pharmacology) Objectives and Plans—1982-1983

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OBJECTIVES:

1. To develop a behavioral profile of the reinforcing effects of nicotine and acetaldehyde.

   a. To find a ratio(s) of acetaldehyde to nicotine that will have optimal reinforcing effects.

   b. To examine the potential physical dependence producing properties of acetaldehyde.

   c. To determine if congeners of acetaldehyde have reinforcing effects.

(Product with the most desirable smoke properties, natural tobacco flavors, new and modified brands)

2. Examine other behavioral interactions between nicotine and acetaldehyde using drug discriminating techniques and intraventricular administration techniques.

(Natural tobacco flavors—physiological response)

3. Develop methods for differentiating between the physical (physiological) dependence producing properties and the behavioral dependence producing properties of reinforcing agents.

(Product with the most desirable smoke properties, new blend concepts—defensive advantages)

4. Identify neuroanatomical sites that mediate the behavioral disruption induced by intraventricular nicotine administration.

(Product with the most desirable smoke properties)

5. Examine the interaction of behavioral and metabolic tolerance induced by chronic nicotine administration.

(Product with the most desirable smoke properties, natural tobacco flavors)

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6. Develop a behavioral profile of nicotine induced tolerance using cross tolerance methods.

(Product with the most desirable smoke properties, natural tobacco flavors)

7. Initiate biochemical research projects relevant to the behavioral effects of nicotine and acetaldehyde.

(Product with the most desirable smoke properties, natural tobacco flavors, new and modified brands)

1. To develop a behavioral profile of the reinforcing effects of nicotine and acetaldehyde,we are using a 24-hour self-administration procedure. The animals are first catheterized and then allowed 7-10 days of recovery. After this, the animals are placed in separate chambers and provided access (intravenous infusions) to various compounds. To establish the reinforcing effects of nicotine and acetaldehyde, we employ several control procedures. First, we use a vehicle substitution procedure; second a lever reversal, and third automatic programmed infusion procedure. If a compound functions as a positive reinforcer, we generate dose effect curves, self-administration time course data, and manipulate the work requirement (the ratio size). These manipulations allow comparisons of the reinforcing effects of a variety of compounds.

To gain information about the neurochemical correlates of nicotine and acetaldehyde self-administration, we are examining the effect of altering three neurochemical systems. The animals are maintained under standard self-administration procedures (24 hr. access to the compound) and following stabilization of the behavior (lever pressing), we introduce our chemical manipulation. The neurochemical systems we are manipulating are: nicotinic, catecholamine, and the endorphin/enkephalin. This series of experiments represents an ongoing project designed to develop a behavioral profile of nicotine and acetaldehyde. Data collection on a number of animals should be complete in the second and third quarters.

1.A. Since both acetaldehyde and nicotine are reinforcing agents and each are contained in smoke it becomes important to determine ratio of acetaldehyde to nicotine which produce maximal reinforcing effects. This requires 4-12 rats depending upon the variability. We will hold nicotine constant (16 ug/kg/injection) and vary the acetaldehyde (2,4,8,16,32,64,128... ug/kg/injection). Each dose will be presented for a minimum of ten days or until the behavior stabilizes. This will allow us to determine the optimum ratio of acetaldehyde to nicotine that maintains the most behavior.

1.B. Some substances that are self-administered and function as reinforcers when chronically administered produce physical dependence. We have already demonstrated that nicotine does not produce physical dependence. This question is of particular interest with respect to acetaldehyde because nicotine and acetaldehyde interact with each other in a reinforcing system. Our plan is to

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train rats to lever press for food under a fixed ratio 32 schedule. The food access periods will be 15 minutes in duration spaced four hours apart. When responding has stabilized, the rats will be catheterized and acetaldehyde will be chronically infused 24 hours per day for 20 days. After 20 days of chronic acetaldehyde administration saline will be substituted for acetaldehyde and the animals will be observed for signs of withdrawal. Changes in the ratio performance will be used to identify a withdrawal syndrome.

1.C. To increase our knowledge about the reinforcing effect of aldehydes in general, we will begin to test congeners of acetaldehyde. The animals will be maintained under standard conditions and two methods will be used to identify the possible reinforcing effects of the congener. First, a direct substitution of the congener for acetaldehyde and second, a fading procedure in which a combination of acetaldehyde and the congener will be presented together and acetaldehyde systematically reduced.

2. Since acetaldehyde and nicotine interact to enhance the positive reinforcing effects of each other it is of interest to determine if this interaction is specific to a reinforcer process or extends to other behavioral effects of the two drugs. Two tests are being used to identify other behavioral interactions, discrimination and intraventricular (IVT) administration. Nicotine at doses of 0.05 and 0.1 mg/kg produce partial (30-50%) nicotine correct responding in rats trained to discriminate nicotine from saline. Presession injection of various agonist and antagonists will alter the effect of the low dose of nicotine. This same procedure will be used with acetaldehyde. Presession injections of acetaldehyde will be used to test for agonist or antagonistic effects. A similar procedure can be used with the effects of IVT nicotine. A detailed profile of the effect of IVT nicotine has been developed and can be used to determine acetaldehyde-nicotine interactions.

3. Methods for differentiating between physiological and behavioral dependence to a drug have not been developed. Therefore, our first efforts will be to explore methods for disassociating these two events. We are using a yoked control technique. In this situation, one animal will self-administer nicotine, that is it will have experience with both the positive reinforcing and the pharmacological effects of nicotine. A second animal will receive yoked injections, that is, it will receive only the pharmacological effects. The animals will be run 24 hours per day. During each session food availability will be signaled by the illumination of the three lever lights. Food will be available under a fixed ratio 32 schedule, for 15 minutes six times each day. After several days of chronic nicotine exposure and after the food maintained behavior stabilizes, saline will be substituted for nicotine. Changes in the scheduled controlled behavior will be used as an index for either the pharmacological effect of nicotine termination (yoked animal) or the combined pharmacological and behavioral (positive reinforcer) effects of nicotine termination. This procedure is unique and unprecedented and may take a considerable amount of effort. We expect to complete the first series of manipulations in the third quarter of 1983.

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4. The behavioral effects of intraventricularly (IVT) administered nicotine have been well documented. However, little information is available about the brain site(s) responsible for these effects. Identification of the neuroanatomical sites that mediate the effect of IVT nicotine will help to determine how nicotine produces its behavioral effects. Thirty rats will be trained to lever press for food under a fixed ratio 32 schedule. When the lever pressing has stabilized all the animals will be implanted with an indwelling intraventricular cannula implanted in the following areas: dorsal hippocampus, locus ceruleus, reticular formation, lateral hypothalmus lateral septum and the vestibular nucleus. Each rat will then be tested as follows: A. Five ug of nicotine in the lateral ventricle. B. 0.25 ug nicotine in the brain site. C. 5.0 ug lidocaine in the brain site (lidocaine acts as a sodium conductance inhibitor and produces a reversible lesion of the area it is injected into) and D. 5 ug lidocaine infused into the brain site followed five seconds later by 5.0 ug of nicotine infused into the lateral ventricle. With these procedures we will be able to determine if the brain sites are, first responding to direct application of nicotine and second, involved in the mediation of the effects of IVT nicotine on behavior maintained under fixed ratio schedule of food presentation. Since only ten rats can be tested in a group, we expect we will have results in all four quarters of 1983.

5. Tolerance to the behavioral effects of nicotine in animals following repeated administrations is well documented. Our research is designed to examine the relative contributions of metabolic and behavioral influences on tolerance development. Whether behavioral factors are important in the development of tolerance to nicotine is unknown.

To address this question, two groups of rats (n = 7/group) are responding under a fixed-ratio 32 (FR32) schedule of food presentation. Following stable day-to-day performance and the determination of acute (one or two injections per week) d-nicotine dose-effect functions, single daily injections of nicotine will be administered (chronic administration phase). The dose of nicotine to be administered chronically will be determined from the acute dose-effect function as one which produces a marked (>50%) reduction in the noninjection control frequency of reinforcement. One group of rats will receive the chronic dose of nicotine before daily test sessions (the Before group) and the other group of rats will receive chronic nicotine after daily test sessions (the After group). Daily nicotine administration will continue until the Before group no longer shows behavioral disruption (i. e., tolerance), a period estimated to take from 2-4 weeks. Following the development of tolerance to nicotine in the Before group, the After group will receive the chronically administered dose of nicotine before the experimental session to determine if tolerance has also developed in this group.

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With the Before-After paradigm both groups of rats will receive exactly the same quantity of nicotine on a day-to-day basis. What will vary, however, are the nicotine-induced behavioral alterations experienced by the two groups of animals. The Before group will experience nicotine-induced disruption of FR performance, including loss of reinforcement, while the After group will not. If loss of reinforcement is a critical factor in the development of tolerance to nicotine, then the After group is not expected to show tolerance when, or at least not to the degree that, the Before group does.

If the After group shows no evidence that tolerance to nicotine developed, then the chronic nicotine dosing regimen will be repeated for this group, only this time nicotine will be administered before the daily test session until tolerance develops. The time course for tolerance development here can be compared to that obtained with the original Before group to further verify that the experience of nicotine-induced reinforcement loss is an important mediating factor. Additionally, since tolerance implies that a shift in the dose-response function to the right has occurred, additional higher doses of nicotine will be administered to both groups. Finally, to examine the persistence of nicotine tolerance, daily injections will be discontinued and the dose used to establish tolerance initially will be administered at one to two week intervals to see if the original acute effect is recoverable. We expect this study to be complete in the first quarter of 1983.

6. Cross Tolerance Between (-)-Nicotine and Pharmacologically Related Compounds

Cross tolerance between two substances exists when the establishment of tolerance to one substance results in tolerance to the other substance. We are currently examining whether rats which are tolerant to the behaviorally disrupting effects of (-)-nicotine in FR 32 food-maintained responding exhibit a cross tolerance to a variety of other compounds.

Two groups of rats (n = 5 to 6/group) are being used in this study. One group will be made tolerant to nicotine by receiving daily presession injections as described in the preceding section, and will be maintained on this treatment regimen. The other group will receive daily presession injections of physiological saline. Once or twice per week a test compound will be substituted for nicotine in the tolerance group and for saline in the nontolerance group in order to determine whether a cross tolerance exists between nicotine and the test compound.

Compounds to be tested include a selection of the nicotine analogues previously examined in the discrimination and self-administration paradigms. It is of particular interest to determine the degree of correspondence among these three paradigms for identifying nicotine-like activity. As one further means of determining the specificity of cross tolerance in nicotine-tolerant animals, the use of "reference" compounds which have been widely studied for their behavioral and neuropharmacological effects would be desirable. Reference compounds producing some effects similar to those of nicotine (*e.g.*, CNS stimulants) as well as those with opposite effects (*e.g.*, CNS depressants) would be most informative.

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7. Biochemical Research Projects Relevant to the Behavioral Effects of Nicotine and Acetaldehyde

The development of an understanding of the neurochemical effect of nicotine and acetaldehyde is essential to any explanation of their behavioral mechanism. Two basic questions will be addressed in collaboration with Project 6902.

A. Regional distribution of radiolabeled compounds.

B. What is the metabolic fate of acetaldehyde?

SUMMARY

	Time
1. To develop a behavioral profile of the reinforcing effects of nicotine and acetaldehyde	First Quarter
A. To find a ratio(s) of acetaldehyde to nicotine that will have optimal reinforcing effects	Third Quarter
B. To provide the potential physical dependence producing properties of acetaldehyde	Fourth Quarter
C. To determine if congeners of acetaldehyde have reinforcing effects	Fourth Quarter
2. Examine other behavioral interactions between nicotine and acetaldehyde using drug discriminating techniques and intraventricular administration techniques	Second Quarter
3. Develop methods for differentiating between the physical (physiological) dependence producing properties and the behavioral dependence producing properties of reinforcing agents	Third Quarter
4. Identify neuroanatomical sites that mediate the behavioral disruption induced by intraventricular nicotine administration	First - Fourth Quarters
5. Examine the interaction of behavioral and metabolic tolerance induced by chronic nicotine administration	First Quarter
6. Develop a behavioral profile of nicotine induced tolerance using cross tolerance methods	Third Quarter

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W. F. GANNON

PHILIP MORRIS U. S. A.
INTER-OFFICE CORRESPONDENCE
RICHMOND, VIRGINIA

To: Dr. R. B. Seligman
Date: September 8, 1976

From: K. S. Burns

Subject: Status of PM Alkaloid Reduction Process Studies

Over the past several years we have had an "on again-off again" program in the area of alkaloid reduction. This work has shown that it is relatively easy to reduce the alkaloid level in cut filler below the 0.2% level by batch treatment in a steam/ammonia atmosphere. Time and temperature for such treatment is in the range of 30 minutes/250°F. Our efforts to make this process continuous in something like a rotary cylinder have only been partially successful due to the residence time requirements and problems in containing the ammonia in such a unit. However, this continues to be a longer range objective.

Currently, we are evaluating a new, properly-designed batch unit which should permit an effective optimization of the process conditions for scale-up to a practical production operation. One of the first objectives of this work will be to evaluate the effect of various conditions on product subjectives.

We are also planning to develop a process for recovering the alkaloid, preferably in a marketable form, to alleviate any effluent problems. Even though we have some ideas in this area that we think should work quite well, with a good possibility of patent coverage, this phase of the process could require considerably more development than the alkaloid extraction step.

We are confident that with a moderate R&D effort in this area, a good, commercial process will be forthcoming. And, as indicated by Art Palmer's patent assessment, existing patents should not be a major problem. Nevertheless, I would think it would still be worthwhile to assess the Tamag (Egri) process and weigh the cost to PM, particularly if there is a good possibility that such a process will be required in the near future.

KSB:jl

cc: W. F. Gannon
A. I. Palmer
F. V. Utsch

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PHILIP MORRIS U. S. A.

INTER-OFFICE CORRESPONDENCE

RICHMOND, VIRGINIA

Mr. L. F. Meyer — DATE: October 11, 1977

FROM: P. N. Gatvin

SUBJECT: Increased Nicotine in Smoke

As a follow-up to my memo of 9/21/77 entitled "Increased Nicotine in Merit® Low Delivery Cigarettes", I am enclosing a survey conducted by Ellen Moore of in-house work on methods for increasing nicotine in smoke. The addition of nicotine citrate to the filler appears to have been the primary method for increasing nicotine in smoke. Some work has been done with nicotine pectate in sheet form, blended with filler as a means of varying nicotine delivery in smoke.

P. N. Gatvin

cc: Dr. E. F. Grabow
Mr. L. F. Filson

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IMAGE

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HAROLD BURNLEY
RELIANCE DOCUMENTS

Memo from E. Chambers to V. Willis Re: Cigarettes for Special Study (6/17/98)

Park 500 Process 30-Day Material Balance (7/15/96)

Letter from Helmut Gaisch to R.B. Seligman (9/12/83)

Report by I.L. Uydess & R.L. Dunn, "Mixed Culture Dentrification: Routine Storage and Start-up Procedures for Refrigereated, Denitrified Seed" (8/29/83) (1000417476-1000417492)

Report by I.L. Udyess, "Project 1904 - Annual Report" (4/22/83) (1003180085-1003180123)

Memo from K.H. Cho Re: Project Cancellation - N.O.D. Plant - Park 500 (4/27/83) (2060529739)

Memo from J.C. Gomes to H.G. Burnley, Jr. Re: Status Report - N.O.D. Project (3/19/82) (2022201031-2022201038)

U.S. Patent No. 3,420,241, Method of Preparing a Reconstituted Tobacco Sheet Employing a Pectin Adhesive (1/7/69)

U.S. Patent No. 3,353,541, Tobacco Sheet Material (11/21/67)

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PHILIP MORRIS U.S.A.

INTEROFFICE CORRESPONDENCE

Richmond, Virginia

To: Vivian Willis

Date: June 17, 1998

From: Elizabeth Chambers

Subject: Cigarettes for Special Study

Data are given for the five cigarette samples submitted for a special study.

	Marlboro Lights KS	Marlboro Lights KS Configuration With 24% RLTC	Marlboro Lights KS Configuration With 24% RL150B
Smoke (FTC)			
Butt Length, mm	34	34	34
Tar, mg/cigt.	10.7	10.4	10.3
Nicotine, mg/cigt.	0.80	0.77	0.76
Puffs/cigt.	7.9	7.8	7.9
Tar/Puff, mg	1.3	1.3	1.3
CO, mg/cigt.	12.1	12.1	11.8
pH	5.30	5.30	5.30
NH_3, µg/cigt.	16.4	8.9	13.1
Cigarette			
Total RTD, mm of H_2O	118	122	121
Length, mm	84.0	84.1	83.9
Circumference, mm	24.8	24.8	24.8
Paper			
Permeability, CORESTA Units	25	26	27
Filter			
RTD, mm of H_2O	98	98	98
Length, mm	27.1	27.1	26.9
Tipping Paper Length, mm	31.0	31.0	31.0
Ventilation, %	19	20	21
Filler			
Total Alkaloids, % (DWB)	1.97	1.98	1.96
Total Red. Sugars, % (DWB)	8.1	8.0	8.0
Reconstituted Tob., %	19	24	25
Expanded Stems, %	2	3	2
Expanded Tob., %	11	11	10
BBQ, %	1	None	1
Lamina, %	67	61	62
Wt. of Tob., g (12.5% OV)	0.675	0.678	0.680
Rod Density, g/cc	0.243	0.243	0.243
Rod Length, mm	56.9	56.9	57.1
pH	5.79	5.66	5.75
NH_3, % (DWB)	0.36	0.18	0.30
Humectants			
PG in Filler, %	0.9	0.9	1.0
Glycerin in Filler, %	2.6	2.7	2.7

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	Marlboro Lights KS Configuration With 24% RL (no ingredients added)	Marlboro Lights KS Configuration With 24% RL (no ingredients added, no casing, no A/C)
Smoke (FTC)		
Butt Length, mm	34	34
Tar, mg/cigt.	10.0	9.4
Nicotine, mg/cigt.	0.78	0.82
Puffs/cigt.	7.4	7.4
Tar/Puff, mg	1.3	1.2
CO, mg/cigt.	11.0	10.2
pH	5.22	5.31
NH_3, µg/cigt.	7.9	6.7
Cigarette		
Total RTD, mm of H_2O	112	119
Length, mm	84.0	84.0
Circumference, mm	24.7	24.8
Paper		
Permeability, CORESTA Units	26	25
Filter		
RTD, mm of H_2O	97	98
Length, mm	27.1	27.2
Tipping Paper Length, mm	31.0	31.0
Ventilation, %	21	24
Filler		
Total Alkaloids, % (DWB)	1.94	2.20
Total Red. Sugars, % (DWB)	7.5	6.0
Reconstituted Tob., %	23	22
Expanded Stems, %	3	3
Expanded Tob., %	14	15
BBO, %	1	1
Lamina, %	60	60
Wt. of Tube, g (12.5% OV)	0.681	0.668
Rod Density, g/cc	0.245	0.240
Rod Length, mm	57.0	56.8
pH	5.57	5.55
NH_3, % (DWB)	0.16	0.18
Humectant/Plasticizer		
PG in Filler, %	0.6	ND
Glycerin in Filler, %	2.2	0.7

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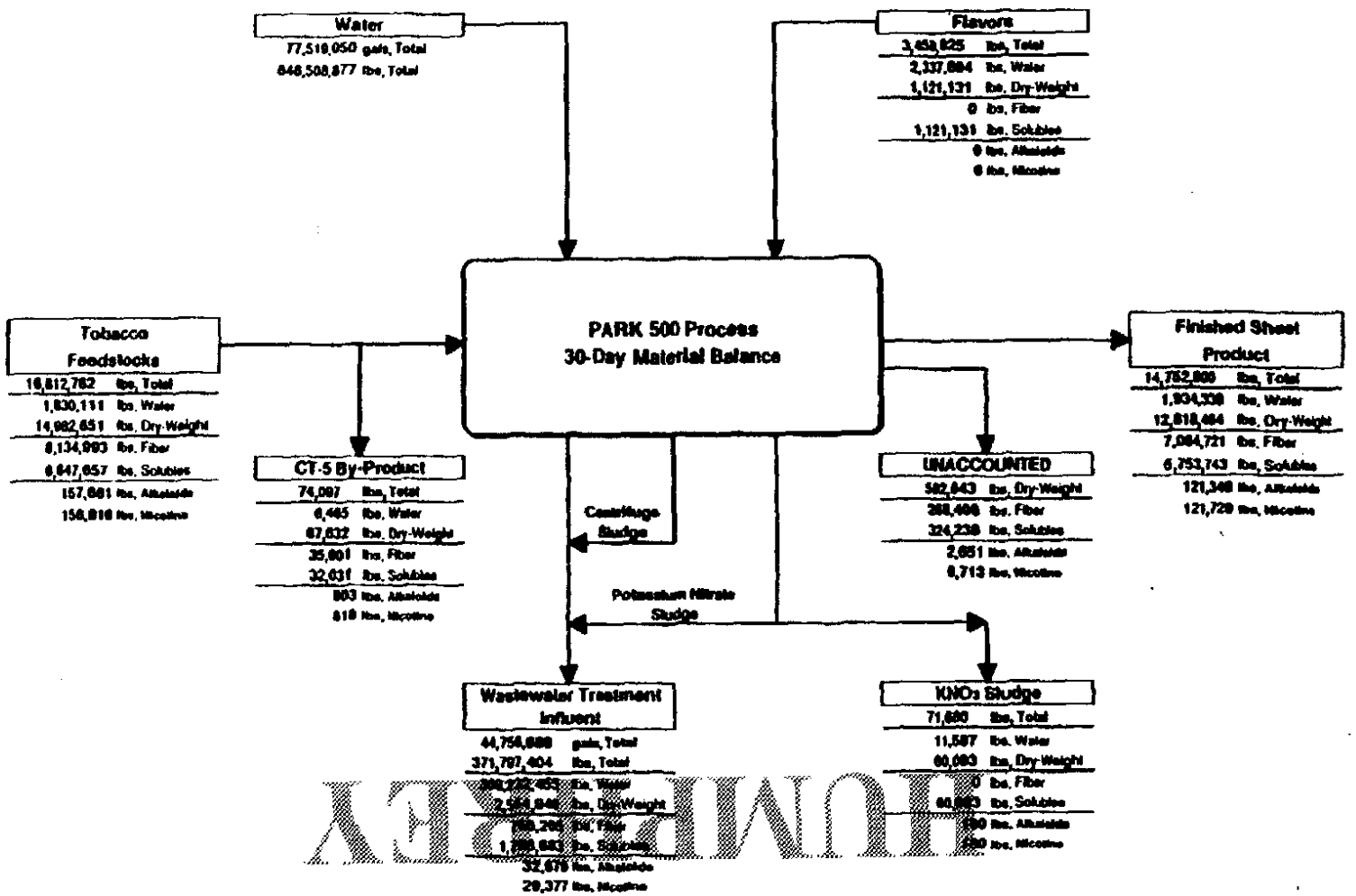

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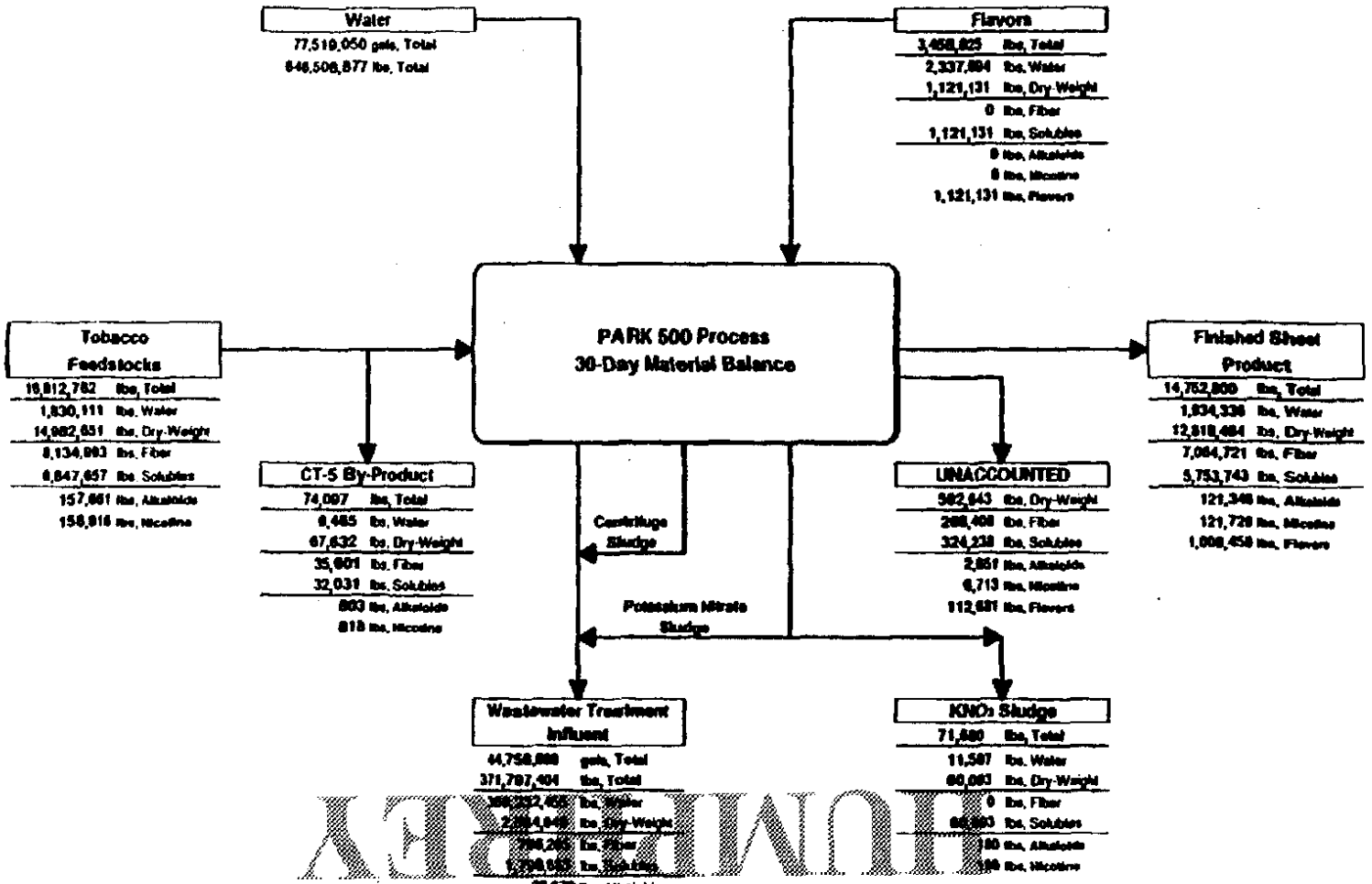

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Based of Park 500 Production from August 23 to September 21, 1995

(lbs, dry-weight)	FEEDSTOCKS	FLAVORS	PRODUCT	LOSSES	% LOSSES
TOTAL	14,982,651	1,121,131	12,818,464	3,285,317	20.4%
TOTAL w/o Flavors	14,982,651		11,810,014	3,172,636	21.2%
COMPOSITIONAL BREAKDOWN					
FIBER	8,134,993		7,064,721	1,070,272	13.2%
SOLUBLES	6,847,657	1,121,131	5,753,743	2,215,045	27.8%
FLAVORS		1,121,131	1,008,450	112,681	10.1%
W/O FLAVORS	6,847,657		4,745,293	2,102,364	30.7%
ALKALOIDS	157,661		121,348	36,313	23.0%
NICOTINE	158,816		121,729	37,088	23.4%
KNO_3	1,047,842		412,131	635,711	60.7%
"KNO_3 DISCARD"			635,711		
VALUES ADJUSTED FOR "KNO_3 DISCARD"					
W/O FLAVORS	6,847,657		5,381,004	1,466,653	21.4%
ALKALOIDS	157,661		123,255	34,406	21.8%
NICOTINE	158,816		123,636	35,180	22.2%

Value based on historic feedstock data for potassium nitrate.

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Park 500 Process - 30-Day Material Balance

Category (lbs, dry-weight)	INPUTS		OUTPUTS				DELTA
	FEEDSTOCKS	FLAVORS	PRODUCT	CT-5	KNO_3 SLUDGE	WWT INFLUENT	INPUTS-OUTPUTS
TOTAL	14,982,651	1,121,131	12,818,464	67,632	60,093	2,564,949	592,643
SOLUBLES	6,847,657	1,121,131	5,753,743	32,031	60,093	1,798,683	324,238
FIBER	8,134,993	0	7,064,721	35,601	0	766,265	268,406
ALKALOIDS	157,661	0	121,348	803	180	32,679	2,651
NICOTINE	158,816	0	121,729	818	180	29,377	6,713
WATER	*1,830,111*	*2,337,894*	*1,934,336*	*6,485*	*11,587*	*369,232,455*	*-367,017,037*
						Process Water Input	*646,508,877*
						Water Delta	*279,491,840*

	% YIELD				ACCOUNTABILITY
	PROD/FEED	FF-PROD*/FEED	WASTE**/FEED	FLAV OUT*/FLAV IN	OUTPUTS/INPUTS
TOTAL	85.6%	78.8%	17.5%	89.9%	96.3%
SOLUBLES	84.0%	69.3%	27.1%	89.9%	95.9%
FIBER	86.8%	86.8%	9.4%		96.7%
ALKALOIDS	77.0%	77.0%	20.8%		98.3%
NICOTINE	76.6%	76.6%	18.6%		95.8%

* Flavor Free PROduct adjusted for flavor content: comparable flavor level = 7.87%

** WASTE Includes KNO_3 Sludge and Wastewater Outputs

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RICHMOND, Va. 23261 / USA

HGA/jig

12th September, 1983

CB3-09190

Dear Bob,

Thank you for your letter, in particular the first paragraph.

I had a good look at the NOD paper which contains a lot more information than had been compiled before. In the introduction the authors say for example "A complete understanding of the relationship between the relative efficiency of denitrification and the variability of our SEL's lies somewhere in the future". I think this sums it up. There are many conditions outlined which must be observed, but they are not terribly well defined and seem, to me at least, not easy to be controlled experimentally.

The most important feature of any industrial process is its controllability. Parameters which spring to mind are temperature, p_H, specific nutrients, etc. I know that the process runs, and I know that biochemically the interactions between two cultures are rather complicated. Where I see a problem, but I might be wrong, is how to keep the process on the rails. For example, what do you do if

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the start-up does not happen or what do you do if suddenly you accumulate the wrong metabolites ? However, the people who work with the system must have a feeling of how well they can control it. They are the only ones who can really judge.

I am asking myself why the process is conceived to be running in the fed-batch mode. The system is obviously fairly sensitive to fluctuations in the composition of the medium. In order to minimise this adverse influence, it was decided to leave a 30 percent inoculum level for re-starting. P_H control, the exclusion of oxygen, and so on have to be done on the remaining two thirds of the (fresh) medium in order to guarantee continuity in the wanted metabolic fashion. If, on the other hand, one would run on the continuous mode, wouldn't the corrections be only minimal and wouldn't the fluctuations be smoothed out ? At the same time p_H control and essential nutrient adjustment (additives) would give one a certain leaverage to steer the process down the right lane. Also in terms of p_{Oxygen} there should be an advantage, because once having reached the low level necessary for anaerobic metabolisation, the partial pressure would never rise again and could certainly not fluctuate into dangerous zones.

I hope to see you soon.

Kind regards and best wishes,

Helmut Gaisch

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CHARGE NO. & TITLE:

TYPE REPORT: ○ ANNUAL ○ SEMIANNUAL ○ COMPLETION ⊗ SPECIAL

DATE: August 29, 1983 PERIOD COVERED:

REPORT TITLE: Mixed Culture Denitrification: Routine Storage and Start-up Procedures for Refrigerated, Denitrified Seed.

BY T.L. Uydess R.L. Dunn

APPROVED BY J.K. Uhlaby

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KEYWORDS: MOD, Dissimilatory Denitrification, Nitrate, Nitrite, CEL, SEL, RU, BL, Microbiology, Biotechnology, Tobacco Processing.

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SUMMARY

Straightforward and reliable storage and start-up procedures are described for the NOD mixed culture seed. The denitrifying product from semi-continuous fermentors is collected with a small residual level of Nitrite still present in the effluent (1-200 ppm NO_2^- ; 0 ppm NO_3^-). The collected material is then immediately refrigerated at 0-4°C. The residual Nitrite present at the time of collection is completely reduced to nitrogenous gas by the time the stored seed cools to 15-20°C, the temperature at which active denitrification ceases[1].

The principle advantage of this storage modality is that the storage form itself is the unmanipulated product of this process and requires no extraordinary preparative manipulations. However, unlike a "dry", low maintenance storage technique such as lyophilization, which renders the dehydrated product more-or-less stable, the activity of the hydrated, refrigerated seed changes with time. This change is manifested primarily as a progressive increase (with storage time) in the time taken to completely denitrify fresh SEL once that SEL has been inoculated with the refrigerated seed. Typically, seed stored at 0-4°C for up to two weeks and then used as a 30% v/v inoculum will denitrify that volume of SEL (NO_3^- = 2000 ppm) within 8 to 16 hours under ambient conditions[2]. Seed stored for 6 months, or more, may take anywhere from 30 to 60 hours to denitrify, depending upon the initial viable cell count and quality of the feed SEL. Sparging fermentors with N_2 prior to and after inoculation reduces the "time to start" by 10 to 50%, again depending upon storage age, viable count, and SEL quality. Generally, however, the older the seed, the greater the percent reduction in start-up time with sparging. Thus, the comparatively retarded activity of older seed does not appear to be due solely to a loss in viability with time, but also (and possibly primarily) to a reduced capacity to induce the required, O_2 - sensitive denitrification enzymes in the presence of an increasingly oxygen-saturated environment. Once the initial startup has been completed, however, process operation (with regard to O-rates, cycle times, etc.) is the same for both "young" and "old" seed-initiated reactors.

While refrigeration of "active" liquid product has been shown to be an effective and easily recoverable storage modality for routine use in the short term, a more stable "dry" storage methodology such as lyophilization would be better suited for securing the integrity of the NOD process in the long term.

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[1] The mixed culture continues to respire Nitrate and Nitrite at a very slow rate even at 0-4°C ("maintenance respiration" - see following report).

[2] No measures are taken to drive the system anerobic (by sparging, etc.).

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Introduction:

The complete dissimilatory denitrification of SEL under anaerobic conditions has been consistently demonstrated over recent months in both laboratory (1) and Pilot Plant Fermentors (2) using the mixed culture of microorganisms orginally isolated from similarly denitrified SEL one year ago (3). Thanks to the collaboration of Dr. Walter P. Hempfling, we now know the principle participants to be an ATCC-like, Gram (+) Bacillus (species unknown), and a Gram negative rod, Alcaligenes faecalis var. denitrificans (4). Both organisms are native to soil and are tobacco identical. Figure 1 diagramatically depicts the proposed respiratory and metabolic relationships between these two bacteria. As can be seen, the Bacillus appear to be the primary Nitrate utilizers (and Nitrite producers) while the Alcaligenes are the predominant (sole?) Nitrite reducers.[1] Each of these organisms share several general environmental and physiological requirements which permit them to function and to participate coordinately in this process (as they undoubtedly do in the soil, or on the tobacco leaves in their native habitat). However, each of these organisms also has specific capabilities, requirements and/or sensitivities unique unto itself. An appropriate minimal set of acceptable circumstances must exist for each organism to function successfully with respect to mediating the complete and consistent dissimilatory denitrification of SEL at a "reasonable" rate. When one (or more) of these environmental or nutritive conditions is not right, their adverse impact on each of these organisms, and thus on the normal phenomenology of the NOD process can be observed as either a deminished capacity to convert Nitrate to Nitrite, Nitrite to gas, or both.

Over the past six months we have made significant advances in our understanding of the major operational and process parameters of NOD (5,6). However, one of our greatest problems has been in gaining a better understanding of the critical parameters regarding seed storage and a consistent process start-up. We now know that the metabolic "acceptability" of the substrate is one of the most crucial factors in this process (as it would be in any other). Unlike a pure culture (single isolate) system, in which a single set of nutritive and environmental parameters satisfies the entire homogeneous microbial population, our mixed culture system, composed of at least two significantly dissimilar microorganisms, necessitates that multiple sets of minimally-acceptable environmental and nutritive parameters be provided. Although considerable light has recently been shed on this topic as a result of the recent experimental observations of the NOD laboratory and Dr. Hempfling, et. al., (4) a complete understanding of the relationship between the relative efficiency of denitrification and the variability of our SEL's lies somewhere in the future.

---

1 The Alcaligenes are also capable of reducing NO_3^- to NO_2^-, as well (personal communication from Walter P. Hempfling).

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FIGURE 1

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While an increased level of understanding of these relationships has definately advanced our ability to control and implement our semi-continuous denitrification process, we have not had, until recently, a good handle on the critical parameters necessary for short and long term storage of the mixed culture, nor have we been able to demonstrate repeatably and reliably an efficacious and rapid start-up procedure.

In October, 1982 it became apparent that refrigerated (denitrified) product stored for 15 months at 0-4°C could be used successfully as inoculum to re-start the NOD process in fresh SFL (3). However, the time necessary to "start" was slow (>48 hours) and multiple trials were inconsistent. But this material did start, and it was from this point which we began our study of the optimization of the refrigeration of bulk, liquid product as a short-to-moderate term storage modality.

Remember, one of our principle project goals was to investigate alternative seed storage methodologies and to define effective start-up procedures for the NOD seed (our previous inability to demonstrate controlled process start-ups was one of the most significant shortcomings we had to overcome). As a result, a start-up procedure was needed which would be:

1. Straightforward - involving a minimum of special manipulation or methodology.
2. Dependable - having a high probability of success.
3. Reproducable - that each start-up had similar results.
4. Predictable - that the time-course and phenomenology of each start-up was predictable based upon a large data-base of previous start-up results under widely varying conditions of substrate and seed, and,
5. Rapid - that the start-up procedure of choice allowed for as rapid a start-up as possible within the confines of the microbiological and chemical systems employed.

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Results and Observations:

We began by refrigerating multiple, 2-liter aliquotes of freshly denitrified product for a period of several days to several months. The refrigerated product was then removed from the coldroom at various intervals (representing different durations of storage) and added as a 10% v/v inoculum to sterile SEL feeds adjusted to pH 5.8 to 6.0 and held at 46-47°C prior to inoculation (5). The time to start[1] was generally poor, (even for "fresh" inoculum) and in the range of 36 to 48 hours (or more). However, our continued experience running the mixed culture in both semi-continuous and batch fermentors soon elucidated to us the mixed culture's rather stringent requirements for a minimal sustained level of excess respiratory substrate (Nitrite) during process operation, as well as during collection of product for future seed (1,6,7).

Experiments conducted in the coldroom on freshly-collected (46°C) product containing excess levels of Nitrate and/or Nitrite demonstrated that significant levels of these substrates were respired during cool-down of the culture. Typically, 2-300 ppm of Nitrate/Nitrite could be reduced to gas during the first 1/2 to 2 hours of cool-down, or, until the temperature of the mixed culture was reduced to approximately 25°C.[2] For this reason, the NOD seed was always collected with a residual NO_2^- level of at least 100-200 ppm's (when possible) to avoid loss of this denitrification capacity (re-repression of the dissimilatory denitrification enzymes).

However, simply satisfying the residual respiratory substrate requirement of these organisms alone was not enough to guarantee fast, repeatable and dependable start-ups from refrigerated seed. There was something else.

---

1 The time from inoculation to the complete denitrification of the initial (starting) "batch" volume of feed SEL in the fermentor.

2 Remember, these organisms are native to the soils of many of these temperate tobacco-growing regions where they grow under widely varying climatic conditions.

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Several rather blatent operational mistakes then became apparent to us. Unwittingly, we had been subjecting our stored inoculum to two rather severe environmental shocks during our start-up procedure. Firstly, we were instantaneously introducing cold (0-4°C), dormant seed into a comparatively "hot" environment (46-47°C) and then asking it to "perform". This sudden thermal transition alone was certainly enough to perturb the mixed culture.[1] Above and beyond this, we were also rapidly introducing a small volume of stored seed at a pH of 7.6 to 8.2[2] into a larger, bulk environment of feed SEL at a pH of 5.8 to 6.0, (another significant environmental shock[3]).

Realizing this, we modified our start-up procedures to avoid (as best as possible) both these abrupt changes in temperature and pH (8).

Current Methodology

Denitrified seed, stored for some variable period (days to months) at 0-4°C, is now allowed to warm naturally at room temperature to between 15° to 20°C and is then introduced into a sterilized "cold" volume of SEL feed held at 15° to 20°C. The feed SEL has also been adjusted to pH 7.6 to 8.0 to match the "finishing pH" of the stored seed (see discussion of the relationship between denitrification and pH, reference 6). Furthermore, the percent inoculum used has been increased (from 10%) to 30% v/v. Ten and twenty-percent inoculum start-ups (in air)[4] were observed to be quite variable with regard to the length of time necessary to start. It wasn't until 30% v/v inocula were employed that highly consistent and reliable start-ups were obtained routinely.

---

1 There is a considerable body of literature on the effects of abrupt thermal transitions on the microbial cell cycle. The results range anywhere from mild delays in the timing of cellular events, to cell lysis.

2 Remember, in the NDD process pH rises as denitrification goes to completion.

3 Abrupt and significant changes in pH can have a manifold adverse impact on many macromolecular structures, especially on biological membranes, which ultimately participate in virtually all cell processes.

4 Under ambient conditions, without sparging.

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The observed variability in start-up time using lower volume inocula under non-anaerobic conditions is indicative of a phenomenon in which some minimum population of "competent" cells is required to successfully initiate and complete prerequisite tasks obligatory to this anaerobic dissimilatory denitrification process (e.g. O_2 removal). Oxygen is known to differentially inhibit the synthesis of the enzymes of this pathway, especially of Nitrite Dehydrogenase, which mediates the reduction of NO_2^- to gas (9,10,11).

The adverse impact of oxygen on the progression of denitrification in "batch" systems using small numbers of cells has recently been demonstrated in this laboratory by J.P. Kusel (12,13). These data are consistent with our previously mentioned fermentor data and with the recent mixed culture model of Hempfling, which depicts the Gram Positive population of Bacilli as being the oxygen "scrubbers". It is envisioned that under non-anaerobic conditions, the Bacilli remove (respire) the residual dissolved oxygen thereby making it possible for the Gram Negative Alcaligenes to reduce NO_2^- to NO. Until this function is completed, Nitrate will be converted to Nitrite, but Nitrite will not be removed to gas. Purging the system with N_2, Ar_2 or He_2 drives the environment more anaerobic and alleviates some of the constraints placed on cell number (of the Bacillus sp.) greatly.

Armed with a greater understanding of the relationship between the mixed culture's capacity to denitrify and the state of their transition into and out of that environment, we set out to examine the relationship between start-up time and the "age" of the stored inoculum (storage age at 0-4°C). Approximately 80 "cold" start-up experiments were conducted using 30% v/v inocula under the experimental conditions outlined previously. The results from these experiments are given in Graph 1. The squares (□) indicate data from experiments in which there were known (documented) operational problems or mishaps resulting in artifically-long start-up times (including power failures and temperature controller failures).

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The circles (⊘) represent the 30% inocula, cold start-up data from the Pilot Plant (seed fermentors T-3 and T-4) during the recent NOD test runs of June-July, 1983. As you can see, the fit between "storage age" and the "time to start" is really quite good, especially in light of: 1) the chemical variability of our SELs, 2) the "biological" nature of the system, and 3) the variability in the initial cell number of the seed used (Product collected from actively denitrifying fermentors may contain anywhere from about 1 to 5 x 10^9 cells/ml depending upon the quality and original Nitrate level in the feed SEL). Graph 2 gives the "time-to-start VS seed age" data for equivalent aliquotes of inocula of the same storage age under "as-is" (*——*) and N_2 - sparged (+----+) conditions. In each case, the N_2 - sparged cultures started-up faster, again demonstrating the known requirement for anaerobiosis.

The form of the data for "as-is" (non-anaerobic) start-ups in Graphs 1 and 2 originally led us to believe that start-ups from "older" seed were primarily cell-number limited -- that there was a progressive loss in viability with increasing age of storage at 0-1°C. (That the longer start-up times experienced with "older" seed were a reflection of the increased time required to replace successively larger portions of the microbial population lost during long intervals of storage). However, the normalization of the data for the Nitrogen-sparged start-ups in Graph 2 argues more for re-repression (loss of capacity) of the Nitrite - removing enzymatic machinery than for loss of viability (although there may be a small loss in viability also - but not to the level that we had previously envisioned).

It is interesting to note that the data for all but one of the Pilot Plant start-ups cluster in the low range of start-up times for the ages of seeds used (e.g., the same seed started up faster in the Pilot Plant fermentors than in laboratory fermentors). The only significant difference in conditions between the laboratory and Pilot Plant is the fact that the Pilot Plant SEL is sterilized within the fermentors with clean steam. The steam, which is sparged through the SEL, sterilizes it, but also drives off dissolved gases, such as oxygen, thereby making the Pilot Plant sterilized SEL comparatively more anaerobic. This effect is more prominently observed in "older" seed which may have a greater saturation of dissolved O_2 and a somewhat deminished capacity to deal with it. Once a de novo start-up has been successfully completed and feeding of the denitrified "heel" is begun, normal semi-continuous process operation can be initiated (perpetuated) and the same nominal dilution rates obtained irrespective of the original storage age or start-up time of the seed.

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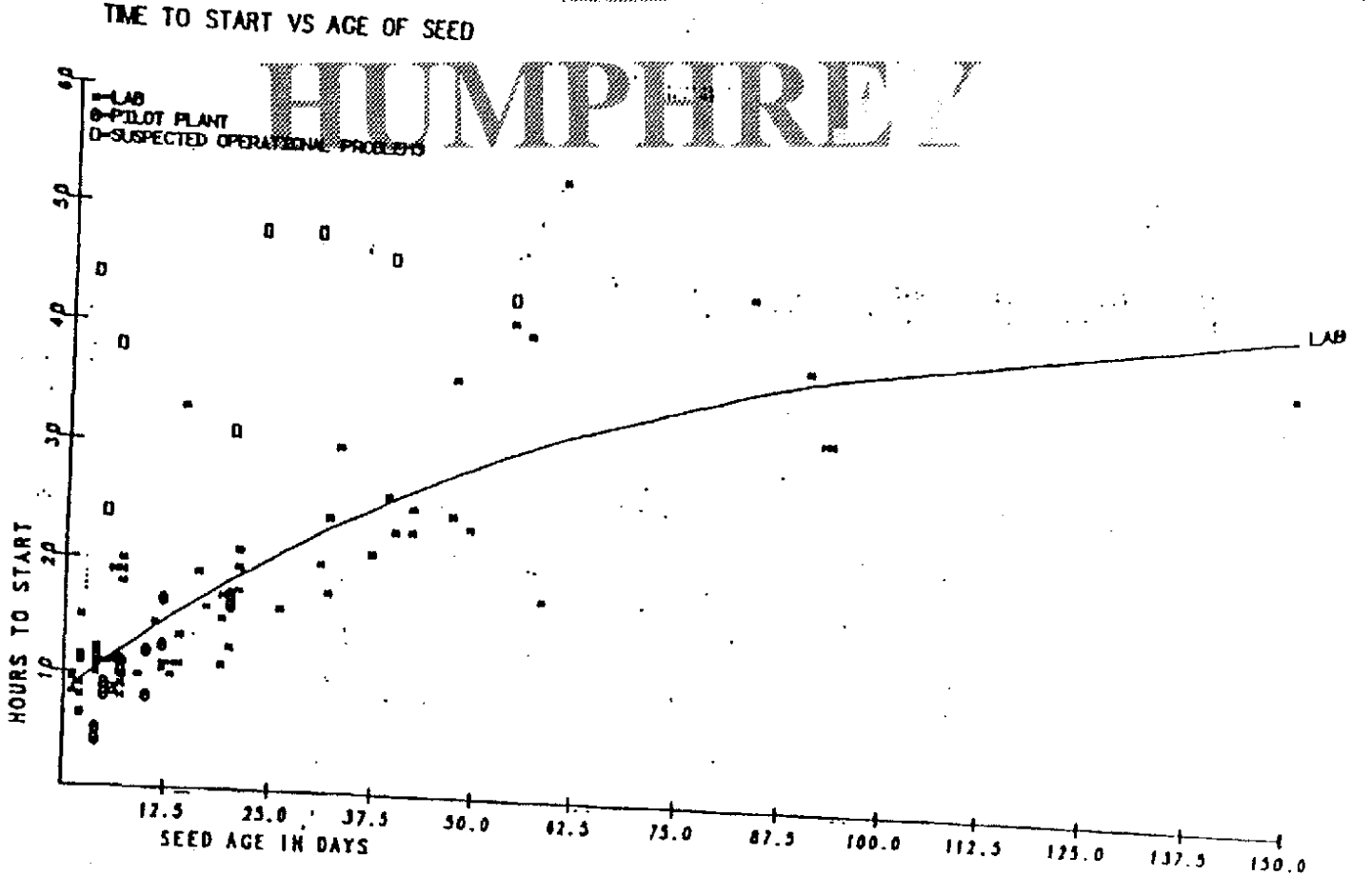

GRAPH 1

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N2 SPARGED VS NON-SPARGED STARTS

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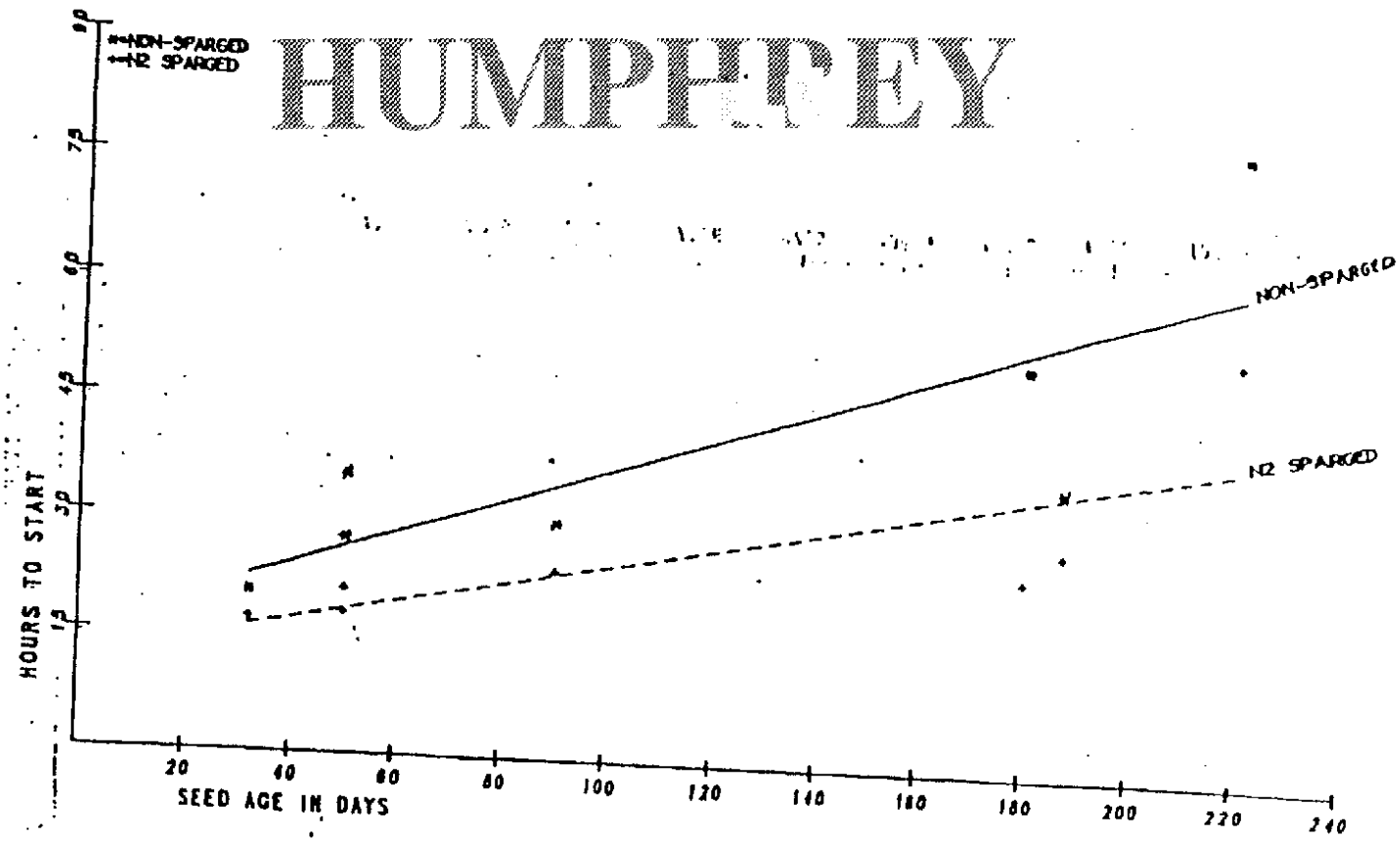

GRAPH 2

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Conclusions:

Now that the major environmental and metabolic parameters of the mixed culture are better understood, we have been able to demonstrate the utility of using the refrigerated, denitrified product from one denitrification run as seed for starting up subsequent denitrification runs. The time required to completely denitrify the original "batch" volume of feed SEL in such de novo start-ups has been shown to be related to the duration of storage at 0-4°C: the longer the duration of storage, the longer the interval to totally denitrify the initial batch volume of SEL.

Generally, under ambient start-up conditions, seed stored for 1 to 2 weeks will take 10 to 18 hours to start, while seed stored for 4-6 months will take 40 to 60 hours to start. Sparging the de novo start-up environment with N_2 results in a 10% to 50% reduction in start-up time, depending upon the original storage age of the seed. (For seed stored up to 6 months, this means a total start-up time of not usually more than 24-36 hours).

Although this storage modality may not prove to be the most efficient vehicle for long-term maintenance of the NCO seed, it does have several important advantages over other procedures.

1. The storage form is the natural (hydrated) product of this process and requires no special or extraordinary processing or manipulation to prepare it either for storage or for use a inoculum.

2. The storage form can be used directly as inoculum to (re)establish active denitrification under non-stringent (ambient) conditions in a rapid, dependable and straightforward manner.

3. The performance (denitrifying potential) of the stored product is known and predictable from: a) the storage age of that refrigerated culture, and b) the past performance of that culture in the fermentor(s) of origin (there is no reason to expect significant deviation from this past performance since this storage modality is not subjected to any perturbatious manipulations).

---

1 In an equivalent "quality" SEL, that is,

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On the negative side, however, is 1) the long-term instability of this hydrated refrigerated material, 2) the requirement to regenerate fresh seed at frequent (monthly) intervals[1], and 3) its' suseptability to spoilage as a result of an interruption in refrigeration. Storage at 0-4°C does not preclude continued chemical activity at both the microbial and environmental (substrate) levels. Long-term Nitrate utilization studies in the coldroom have already demonstrated that the mixed culture will continue to slowly respire Nitrate and Nitrite at 0-4°C (presumably, maintenance respiration). Chemical modifications to the SEL also undoubtedly occur (remember, the pH of the refrigerated material is often in the range of pH 7.8 - 8.0). For this reason, and to otherwise "protect" the integrity of this process in the future (both in the short and in the long-term), a more stable and environmentally independent storage modality is required. In this context, lyophilization would appear to be the most efficatious and logical choice.

In lieu of this, J.P. Kusel has recently demonstrated that small volumes of mixed culture seed that have been successfully lyophilized as 1:10 dilutions of the original product in a defined (non-SEL) medium, can subsequently be used to start laboratory-scale fermentors after an initial re-hydration/re-activation step [14]. A joint program with an outside consultant/contractor will soon be undertaken to optimize this long-term storage modality and to produce certified, lyophilized seed for future NOD development.

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[1] If rapid startups (8-12 hours) are desired.

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This work could not have been conducted without the assistance of the PM Temporary Employees who worked long hours to run and maintain these experiments: P. Finn, P. Boone, A. Conyers and M. Varnier.

Special thanks to H. Hartung for his instruction and continued guidance on the use of his EZ DATA and EZ PLOT programs, and to Pat Finn and Andrei Conyers for implementing these programs.

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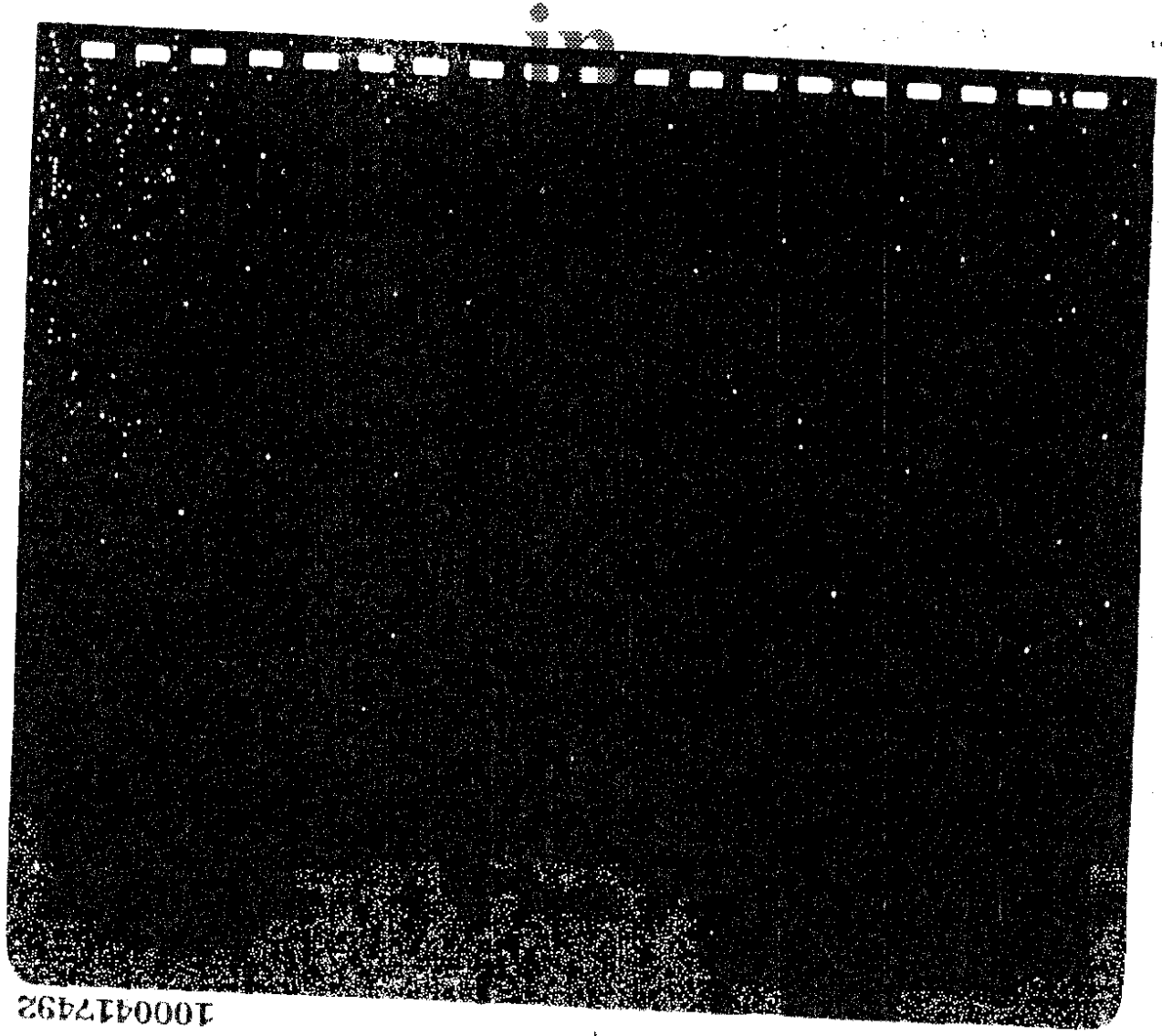

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ABSTRACT

The progress made to date to elucidate the phenomenological basis of, and operating and monitoring parameters for the Naturally Occurring Denitrification process (NOD) is discussed.

Organisms indigenous to tobacco have been shown to be capable of mediating the complete disimilatory denitrification of our strongly extracted tobacco liquors (SEL) at industrially-acceptable dilution rates of >10%. Although considerable effort was expended examining the denitrification potential of a single isolate pure culture (ATCC-1), the mixed population of bacteria originally isolated from our spontaneously-denitrifying SELs has been shown to be more reliable and less fastidious with respect to its use in an industrially-scaled process.

Operational, process, and monitoring parameters are discussed in light of laboratory-scaled fermentation experiments. The optimization of start-up, temporary hold and shut-down procedures is also discussed.

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INTRODUCTION:

An historical overview of the Naturally Occurring Denitrification process (NOD) has previously been given.[1] Although the original observations upon which this process was based were shown to be the result of the activity of a mixed population of bacteria, subsequent process development aspirations re-directed the laboratory effort toward identifying and utilizing a single, pure culture isolate upon which an industrially-acceptable process could be designed. A thermotolerant Bacillus (sp. unknown) isolated from SEL by the American Type Culture Collection and alleged to be a potent solitary denitrifier[2] became the focus of the NOD laboratory investigations. Laboratory and Pilot Plant operating procedures were modified to incorporate "aseptic operation" to accommodate working with the pure culture. However, a number of important questions persisted with regard to: 1) the true purity of the ATCC isolate,[3] 2) the isolate's inherent capacity to function as a potent and solitary denitrifier,[4] and 3) the possible perturbations suffered by the isolate as a result of the methodology employed during isolation and lyophilization at ATCC. Laboratory and Pilot Plant experiments failed to verify conclusively, that the pure culture that was supplied to PM by ATCC was indeed capable of reproducible start-ups and complete denitrification. As a result, our attention turned once more toward the mixed culture from which this isolate had originally been obtained. Subsequent experiments with both the mixed culture and freshly-isolated "pure" cultures of ATCC-1-like strains of Gram + and Gram "variable" bacilli repeatedly demonstrated that 1) the mixed culture was apparently a more reliable and more potent mediator of

1. Banyasz, J., Proj. 1904 Annual Report, May 21, 1982, Accession #82-159.
2. Ibid.
3. Op. cit. p. 3, and recent observations.
4. Uydess, I. L., "Denitrification," memo to J. Banyasz, October 7, 1982.

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complete dissimilatory denitrification than ATCC-1, and 2) that while ATCC-1-like organisms may be capable of complete denitrification under certain conditions, we didn't understand enough about the (unique?) requirements of this elusive capacity to utilize it within the confines of our physical planning to develop, engineer and implement an industrially acceptable and commercially feasible NOD facility for PM USA. In lieu of this, we proceeded with our investigations using the mixed culture. However, several crucial questions still had to be answered:

1. Could the mixed culture be proven to be a reliable and potent denitrifier?,
2. Could straightforward, dependable (predictable) start-ups be demonstrated from "stored" seed?,
3. Would the mixed population remain stable over long periods of growth and denitrification with regard to:
   a. it's microbial composition (population consistency),
   b. it's ability to sustain active denitrification over long periods of time, and,
   c. the quality (variability) of its product?
4. Could an industrially acceptable process be demonstrated (e.g. >10% dilution rates) using the mixed culture? and,
5. Could the mixed culture cope with the variability in quality (composition) of our SELs?

The following report summarizes the progress made in answering these questions over this past year. Portions of this document are abbreviated due to the detailed coverage of some of these topics in recently issued special reports and memos which are referenced or appended herein.

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RESULTS AND OBSERVATIONS:

A. THE BACTERIA:

The Naturally Occurring Denitrification process (NOD) as originally observed and documented by B. Semp[5] at our Park-500 RL facility, was shown to be mediated by a mixed culture of bacteria. Our own light and electron microscopy observations confirmed this, and further elucidated that the mixed population was apparently composed of both Gram (+) and Gram (-) rods of diverse morphologic forms, as well as some Gram (+) cocci and spores.[6] At about that same time, the American Type Culture Collection facility (ATCC) isolated and partially identified four Gram (+) and/or Gram variable bacilli (species unknown), one of which (ATCC-31973; our ATCC-1) appeared to be the predominant denitrifier.[7] However, subsequent investigations within PM R&D were not able to consistently show that this lyophilized pure culture as supplied to us by ATCC was a reliable mediator of the complete denitrification (to nitrogenous gases) of our SELs.[8] An immunofluorescence assay employing specific antibody made to ATCC-1 developed by PM R&D personnel[9,10] did, however, confirm that this bacterial (antigenic) type was a major component within the mixed population. However, such tests as these, although potentially powerful tools, only verify the genetically-specified antigenic "fingerprint" of an organism's surface, and not the physiologic capacity of the organism itself.

5. B. A. Semp, "Exploitation of Naturally Occurring Fermentation," memo to C. H. O'Donohue, Nov. 19, 1980.

6. I. L. Uydess, EM observations, Lab Notebook 7645, 1981.

7. Malik, V. S., personal communication(s), 1982.

8. J. Banyasz, Proj. 1904 Annual Report, Acc. No. 82-159, 1982.

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10. R. W. McCuen, "Production of Specific Antiserum using ATCC-1," Special Report, Feb. 1983, Acc. No. 83-054.

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As our investigations shifted to an examination of mixed-culture mediated denitrification, we recognized the need to verify, as best as we could, the composition and stability of this heterogeneous mixture of bacteria during prolonged periods of growth and denitrification. However, our in-house tools and expertise were not particularly extensive in these areas. As a result, we initially had to rely upon rather limited characterization techniques, including a) light microscopy, b) the Gram stain, c) colony morphology on agar plates, and d) a bank of biochemical test media, all in conjunction with our daily observations and data on the "quality" of denitrification in whatever experimental situation was being examined at the time. However, within the confines of the relatively simple tools at hand, we were able to verify that the overall quality of the mixed culture (with respect to the apparent number of phenotypically-different participants therein) had not changed significantly oven the past four months of more-or-less continuous growth and denitrification in laboratory-scale fermentors. Biochemical, cultural and morphological observations coupled to more recent immunofluorescence assays continue to show the presence of Gram (+) to Gram variable ATCC-1-like species which comprise a major portion of the total bacterial population. Additionally, the Gram stain and various other morphologic, cultural and biochemical tests also demonstrate the continued presence of both Gram negative and Gram-variable rods, and apparently, Gram (+)/variable cocci. Each of these morphologic types have recently been isolated and partially characterized by Dr. Walter P. Hempfling,[11] a collaborator and consultant to the current NOD Bioengineering program. Additional information on the taxonomic identifications and physiologies of these organisms will be made available once the preliminary findings have been substantiated.

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11. Department of Biology, Univ. Rochester, Rochester, NY.

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B. THE PHENOMENOLOGY OF THE PROCESS:

1. Calculation of Equivalent Dilution Rate in a Semi-Continuous System:

Before presenting a description of the process itself, it would be helpful to first set forth and explain how we calculate Dilution Rate (D-rate) in the laboratory. The term "Dilution Rate," as it is used in the bulk of the microbiology literature, primarily addresses itself to a description of the throughput (or fractional volume changes per hour) of a continuous culture system having a fixed (non-varying) total working volume (such as a Turbidostat or Chemostat). The formula for calculating the "D" rate of such a system has conventionally been given as: $D = f/v$ where "f" is the flow rate of substrate per hour (in and out of the system), and "v" is the fixed working volume of the system.[12] This also implies that there is a fixed population of microorganisms - the exact number being defined by the specific growth rate under those conditions of substrate and environment. In this program, however, we knew that we would be dealing with a semi-continuous "fed batch" system in which a given volume of fresh substrate would be added over some defined period of time to "X-volumes" of a previously processed substrate ("heel") containing the active culture of bacteria. This, then, specifies two major departures from classical continuous culture methodology.

First, we would now be dealing with a continuously changing (increasing) working volume, and second, a necessary and more-or-less concomitant increase in the total "working" population of bacteria. Thus, our "black box" isn't constant, but dynamic. (We would, in effect, be adding X-volumes of fresh substrate per unit time to an ever-increasing working volume; dv/dt from T_o to T_{final}). As a result, we decided to modify the classic dilution rate formulation to better suit our operational and functional needs - after all, our primary

12. Herbert, D., et al., J. Gen. Micro. (14) 601-622, 1955.

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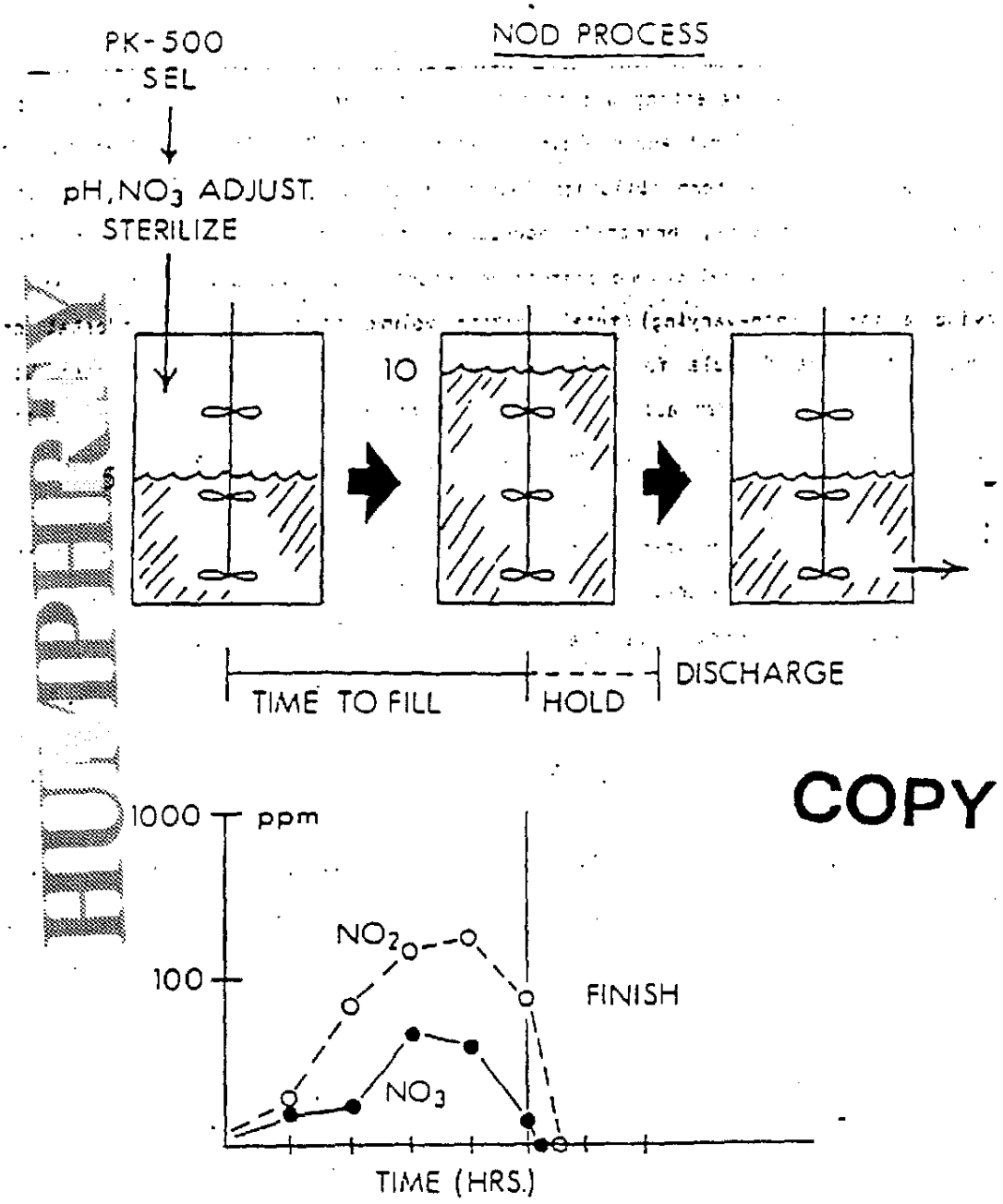

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FIGURE 1

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interest was to most accurately describe our working system. Although this would best be done by integrating these changes in the actual working volume with time, we decided to approximate this by simply using the arithmetic mean filling (working) volume half-way through fill as "v". We rationalize this because we know that "the system" doing the work only attains it's true total working volume when it is finished filling, and in a majority of instances, the work has been completed (or nearly completed) by the end of the fill. Thus, some constantly changing volume (dv/dt) that is almost always less than the final full volume is doing almost all of the work. This is diagramatically represented in Figure 1.

Furthermore, we also know that in other instances, the total cycle time can be longer than just the total filling time (our process isn't always finished exactly when the fermentor is finished filling, thereby requiring a post fill - finishing period). Thus, we also could not simply use the value for the rate of addition of substrate per hour ("f") either. Instead, we settled on the following formulation:

$$\frac{f_t + T_t}{V_{ave}}$$

where f_t is the total volume of substrate added to the fermentor during that fill (e.g., the replacement volume); T_t is the total time for that cycle (fill time + finishing time; if not "finished" by the end of fill), and V_{ave} is the mean full (filling) volume of the fermentor (e.g., volume of the "heel" + half the replacement volume). If the time required to discharge the product were substantial, it too would have to be included in the value for the total cycle time. However, since our laboratory-scale fermentors can be discharged in just a few minutes, we have not included this value in our calculations for "D".

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2. Process Chemistry:

Since we have recently documented in detail both 1) our experience over the past 6 months running the mixed culture in shaker-flasks and laboratory-scale fermentors,[13,14] as well as 2) what we currently understand to be the major operational, process and monitoring parameters of the mixed culture denitrification system[15] (Appendix A, attached), we will outline only the relevant data and observations here. Otherwise, any additional information regarding the specifics of start up and shut down procedures, etc. may be obtained from either the aforementioned or following referenced materials.

One, or possibly more of the bacterial strains in our mixed culture mediates the complete dissimilatory removal of Nitrate-Nitrogen to Nitrogenous gas. The process is thermotolerant and anaerobic. Although some question still persists concerning both the positive and negative influences of micro-quantities of oxygen during the initial start-up (induction) period and throughout normal process operation, no extraordinary steps are taken at this time to exclude oxygen from this system. It will probably be some time (months) before this particular point is more adequately understood. Independent of this, the mixed culture appears to be able to cope with rather wide variations in the "quality" of its chemical and physical environment, as demonstrated by its ability (within limits) to grow in and actively denitrify our Park-500 SELs. Recent experiments with single component SELs produced in the R&D Pilot Plant[16] have helped us characterize one of these substrate quality "end points" with regard to the

13. Uydess, I. L., Memo to J. Banyasz, October 7, 1982.

14. Uydess, I. L., Special Report, April 1983.

15. Uydess, I. L., Memo to J. Whidby, March 31, 1983.

16. Thanks to Grant Gellatly and crew.

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acceptability of the major SEL blend constituents. Specifically, 100% Burley, Bright and Oriental lamina SELs were found to be extremely poor substrates for both growth and denitrification, while 100% Burley and Bright stem SELs were each found to be good substrates for growth and denitrification.[17] These observations will be discussed in more detail in a later section. Although we do not as yet fully understand the physiological (biochemical) basis for the adverse impact that each of these lamina SELs had on the performance of our microbial system, the observed effect was significant and reproducible, and helps explain (and predict) how the blend composition of current and future SELs would impact upon our denitrification process. We are currently collaborating with other R&D personnel to elucidate the biochemical basis (with respect to stem and leaf chemistry) for these observations.

Aside from the positive or negative impact of nominal blend composition upon our system, there also appear to be other chemical and/or physical differences within SELs of the same apparent blend composition which effectively render them either "good" or "poor" substrates for NDD. HPLC and other analytical techniques have not, as yet, yielded discernible information on chemical differences between apparently similar substrates (SELs) which could be directly (or indirectly) correlated to observed changes in the rate of denitrification (and/or growth), thus "throughput" (O-rate) in these substrates. Previous observations on the consistent disappearance (utilization) of certain organic acids such as malic acid during active growth and denitrification have not been made, per se, thus far in these studies, although our HPLC data do show that there are observable differences between the organic acid profiles of various feed SELs and those of the resultant denitrified products. However, we do not as yet know what these compounds are, nor do we understand how their relative presence or absence

17. NDD Labbook No. 7771.

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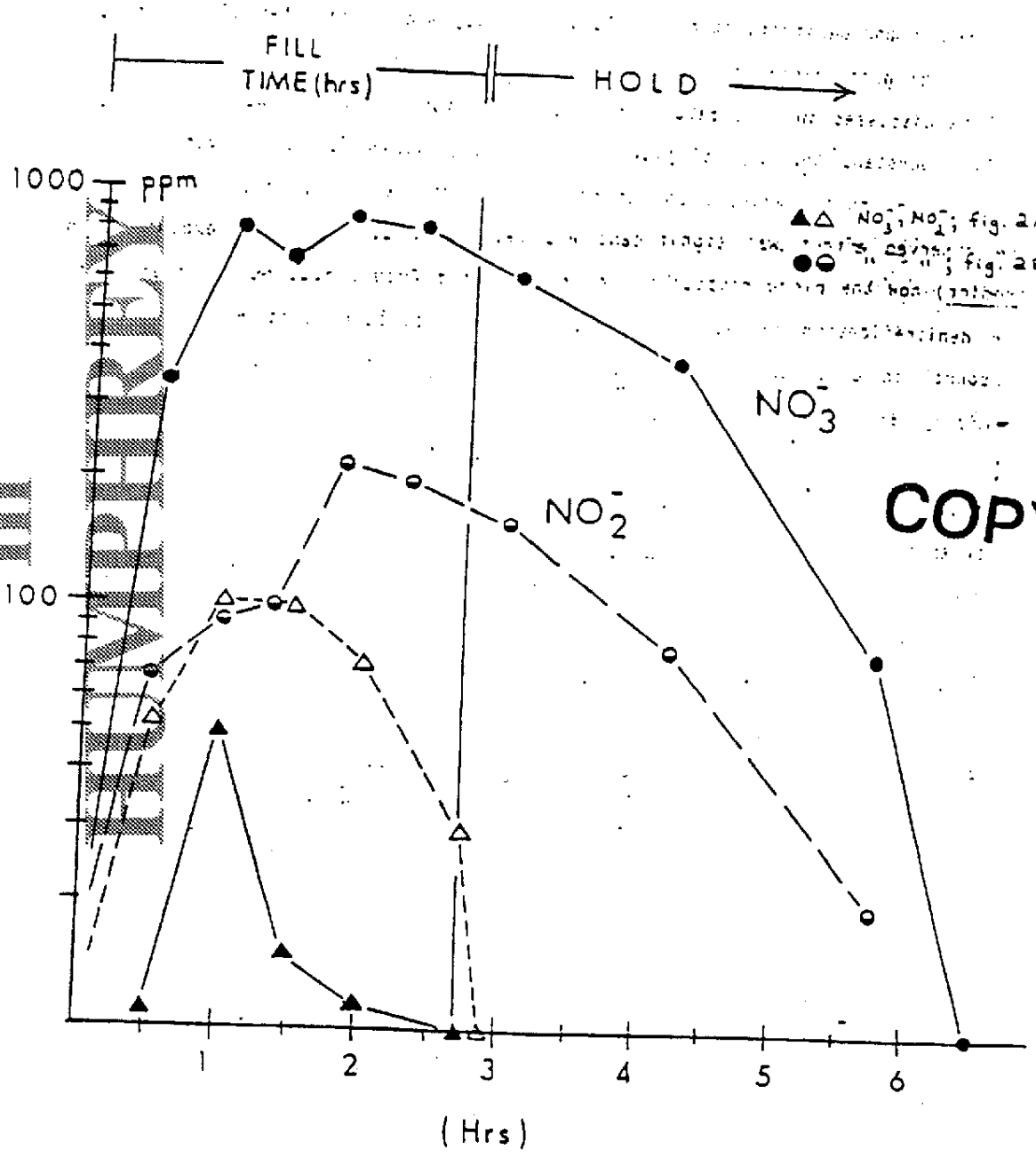
FIG. 2: CYCLE PROPHILES



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affect the growth or denitrification capacities of these organisms. There is, however, the documented observation that the relative quality of the SEL does directly influence the rate of denitrification in that SEL.

Normally, when everything is "right," the incoming feed Nitrate is converted to Nitrite at about the same rate at which the feed is added to the fermentor. Therefore, there is normally no appreciable accumulation of residual Nitrate (NO_3^-) during the fill period (e.g., usually >150 ppm). Nitrite, (NO_2^-), on the other hand, usually accumulates to some low-to-moderate residual level during fill (50-250 ppm), but is then converted to gas (N_2O or N_2) by the end of fill, or shortly thereafter (15-30 min). This is best demonstrated by looking at and comparing individual cycle profiles for Nitrate and Nitrite during "good" (fast) and "bad" (slow) cycles. Figure 2A shows a cycle profile for a typical denitrification cycle during which time the Dilution Rate of the fermentor was between 0.15 and 0.20 hrs^{-1}. Figure 2B, in turn, shows another (later) cycle profile for that same fermentor during a period when the D-rate fell below 0.10 hrs^{-1}. As you can see, the phenomenology of Nitrate and Nitrite removal for these cycles is substantially different, and indicative of a functional problem on the part of the microbial system in dealing with either converting Nitrate to Nitrite, Nitrite to gas, or both. Furthermore, it has also been our experience that a transition from "good" to "bad" cycles occurs most frequently at the time of (or shortly following) the introduction of a new batch (new pick-up date) of SEL into the system (e.g., again, indicative of some qualitative difference in our SELs which has an almost immediate impact on the denitrification capacity of the mixed culture). The rapidity with which this adverse impact on denitrification and D-rate is observed is dependent upon several factors including... 1) the previous "health" and stability of the system prior to that SEL's introduction, 2) the actual amount of that material introduced into the system (the

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concentration of new SEL that the system "sees" during feeding of the 10%, 50% or 75% heel), and 3) the magnitude and nature of the problem with that SEL. For now, it would appear that there are several potential problem areas with respect to the "acceptability" of our SELs:

1) Chemical problems; manifested as either insufficient levels of required metabolic substrates to support the growth and denitrification of that bacterial population,
2) An unfavorable physiochemical environment; which inhibits or interferes with transport and/or energy coupling phenomena, such as the presence of inhibitors of bacterial metabolism and/or respiration, or,
3) The presence of alternative metabolic and/or respiratory substrates; which directly or indirectly interfere with the utilization of NO_3^- and/or NO_2^-.

Although it is difficult to say with certainty which of these is the route of most of our problems, it does appear that the lack of appropriate levels of substrate and/or the presence of alternative (competing) substrate are prime candidates.

However, when the environmental conditions for growth and respiration are favorable, the mixed culture can demonstrate an appreciable aptitude for Nitrate and Nitrite removal. Figures 3 and 4 show the individual cycle times and resultant D-rates obtained running the mixed culture in Park 500 SELs with feed Nitrate levels varying between 2200 and 3500 ppm. As you can see, the mixed culture was able to maintain a dilution rate in excess of 0.1 throughout most of this period of varying Nitrate concentrations. However, on other occasions, when unfavorable SELs were introduced into the system (such as 100% Burley lamina

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FIGURE 1

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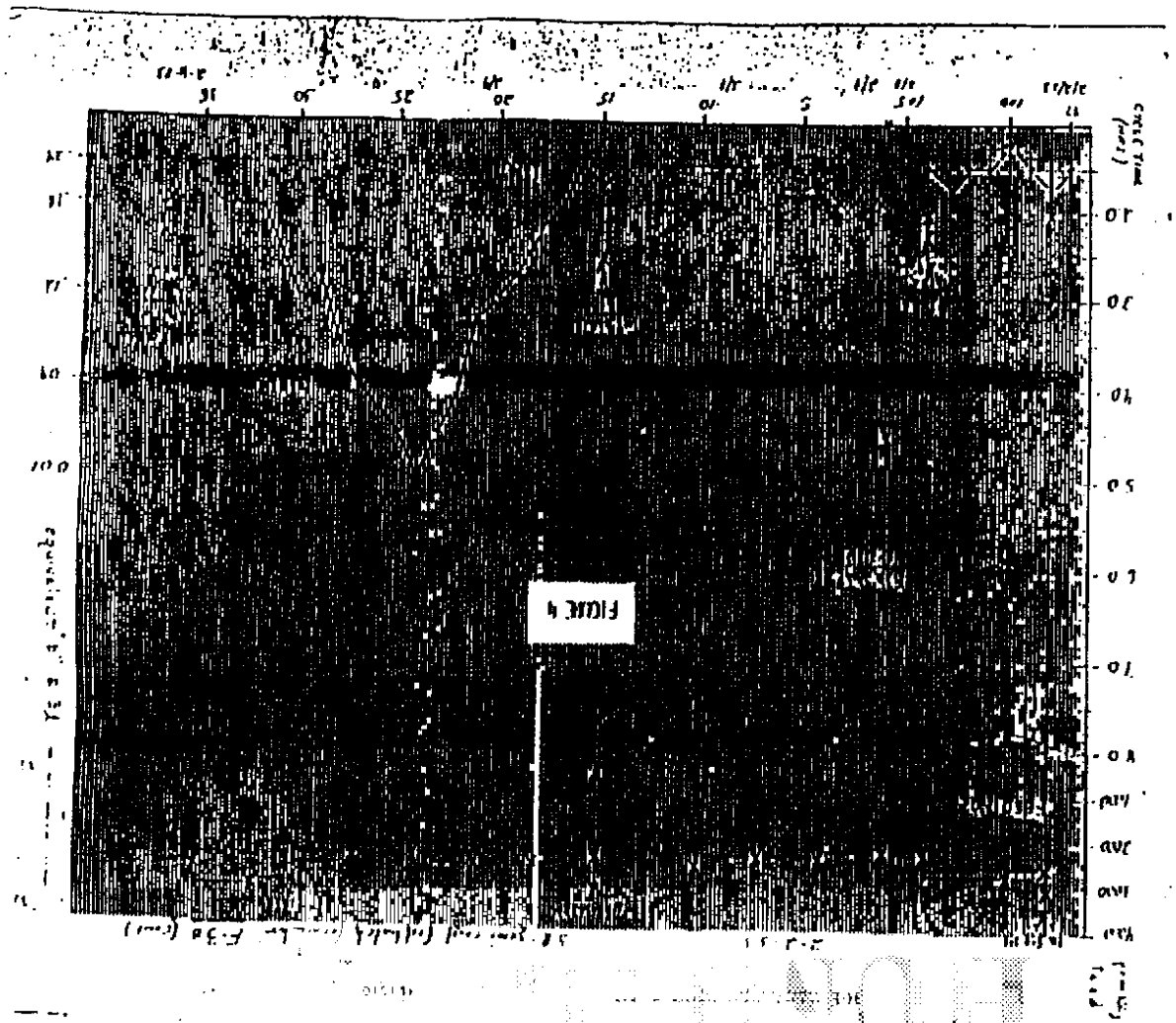

FIGURE 4

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SEL), the O-rate deteriorated sharply soon after its addition to the system (remember, the dilution effect).

An understanding for and appreciation of the magnitude of dependency this system has on the quality of its substrate is crucial to attempts to utilize it in an industrial environment. Our SELs do have the potential to vary in chemistry and day-to-day consistency, and this has a direct effect on the throughput of this system irregardless of how large or how small the operating volumes are. We must endeavor to identify those variable SEL components that are responsible for this biological impact so that we may either; 1) optimize SEL production to avoid (or compensate for) these variabilities, or 2) supplement our SELs with these materials (organic acids?). Furthermore, we also have to be able to predict the operational impact that certain SELs will have on our system prior to their introduction into it. This would also necessitate the testing of future blend changes in a laboratory or Pilot Plant situation as support for the operation of such a facility. These unknown variabilities can have an even greater adverse impact when poor quality SELs are inadvertently used during de novo start ups. Having to cope with the adverse impact of an unsuitable environment is one thing when the culture is "up and running," but having to rely upon it during initial start up from stored feed, is quite another.

3. Nitrate and Nitrite Removal:

Spectrophotometric and GC "headspace" analysis of actively denitrifying fermentors have demonstrated that the feed Nitrate (and resultant Nitrite) are completely removed to nitrogenous gas (N_2O and/or N_2). However, the relationship between which specific off-gas (N_2 or N_2O) is the major or sole nitrogenous off-gas product and the O-rate of the operating fermentor (or initial concentration of Nitrate in the feed) is as yet unclear. We have observed on

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numerous occasions that apparently "equivalent" fermentors (operating at the same D-rate) may have significantly different off-gas profiles with respect to the presence and/or ratio of N_2 and N_2O. Aside from N_2O and N_2, the only other off-gas product of significant concentration (>1%) is CO_2, which appears to predominate (>50% of the total off-gases) when the D-rate is poor (<0.1). This is not particularly surprising since one would probably not expect to find much N_2 or N_2O issuing from a fermentor that isn't removing NO_3^- to NO_2^-, or NO_2^- to gas. Whether GC off-gas profiles can be used to monitor the status of the operating system or predict an impending problem, has yet to be seen. More specific information concerning off-gas profiles for actively denitrifying fermentors as well as the determination of the specific Heat of Reaction for Nitrate conversion to Nitrite, and Nitrite to gas, can be obtained from the references of Watson, D., as well as from that of Martin, P. and Counts, M. E.

4. HPLC and Analytical Data:

Although previous studies[13] reported that Malic Acid was frequently consumed by the mixed culture system during periods of active growth and denitrification, recent HPLC determinations of sterile PK-500 feeds and their subsequent denitrified products have not proven this to be characteristic of the current system. However, we have observed significant fluctuations in the various (including organic acids) peak heights of our pre- and post fermentation HPLC profiles. These encompass both increases and decreases of pre-existing (feed profile) peaks, as well as the appearance of new peaks, presumably the result of the metabolic conversion or degradation of other materials within our feed SELs. Although we have recently increased the spectrum of our known standards (to include Acetic, Propionic, Butyric, Malic, Lactic, etc.), we have not, as yet,

13. See Banyasz, J., Proj. 1904 Annual Report, May 21, 1982, Acc. No. 82-159.

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been able to make any meaningful correlations between the efficacy of denitrification (D-rate) and these pre and post denitrification organic acid profiles. In light of the mixed culture nature of our system, the observed fluctuations in organic acids could easily reflect the complex nature and interrelationship of the various microbial metabolisms being expressed within an equally complex and ill-defined substrate, our SEL. Some of these plant-derived materials in our SELs are most certainly principal metabolic substrates for one or several of these bacteria. However, other microorganisms within the mixed population may well be producing more of these same (and/or different) materials as they metabolize their specific substrates. Hopefully, we will be able to elucidate more about this in the near future as more is learned about the physiologies of the individual participants in this mixed culture of microorganisms.

Data generated in collaboration with the Analytical Division has helped verify the following with regard to the ranges of conditions within which the mixed culture will continue to grow and denitrify in actively denitrifying fermentors:

a. Operating pH:

The "running" pH of actively denitrifying fermentors has been shown to be dependent upon 1) the blend composition of the feed SEL (with respect to its "as-is" pH and buffering capacity), and 2) the original Nitrate level in the feed. The higher the original feed Nitrate level, the higher the resultant "running" and "finishing" pH of the actively denitrifying fermentor. pH with respect to blend composition, is primarily determined by the relative quantities of Bright and/or Burley lamina, and the relative ratios of lamina vs. stem. This relationship between SEL pH and blend composition is known, and has been documented previously (G. Bokelman, 1982). However, there are still certain aspects of the chemistries (buffering capacities, etc.) of our SELs which are

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still unknown, which do appear to have a significant impact upon the mixed culture's ability to grow and/or denitrify within these materials. A better understanding of the nature of this variability in SEL chemistry is paramount to our being able to predict and possibly compensate for how well some SELs will run in our mixed culture denitrification system.

b. SEL Total Solids:

Experiments were conducted to determine if the mixed culture could continue to grow and actively denitrify within SELs having elevated solids levels. To accomplish this, Pk-500 CEL was added back to standard Pk-500 SEL to achieve a total solids level in excess of 15%. These high solids (and high Nitrate) SELs were then fed to an actively denitrifying fermentor running up to that point on 7-8% solids (Pk-500 SEL) feeds. Although the initial addition(s) of this high solids feed to the 50% heel of the fermentor diluted it somewhat, successive 50% volume additions soon elevated the "working" solids level within the fermentor to approximately 14%. The Nitrate levels of these CEL-fortified SELs was also high, and in the range of 3600 to 4000 ppm. The mixed culture continued to grow and actively denitrify these high solids/high NO_3^- SELs up to and including a level of 14% at dilution rates in excess of 10%. However, it is not known whether this can be accomplished within all composition SELs, nor do we know at this point what the longevity of this denitrification capacity would be within such high solids SELs during long term operation. The total alkaloids levels within these high solids SEL feeds were also elevated, and were found to be about 3.0 - 3.5% in the resultant denitrified products.

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5. FEED SEL NITRITE:

Investigations were also conducted to determine the effect of elevated feed Nitrite (NO_2^-) levels on the operational consistency of an actively denitrifying fermentor. To do this, Potassium Nitrite (KNO_2) was added in successively higher concentrations to our SEL feeds prior to sterilization to obtain Feed NO_2^- levels between 200 and 1000 ppm. These Nitrite-fortified SELs were then used to feed an actively denitrifying fermentor. Within forty-eight hours, SEL feeds with Nitrite levels in excess of 600-800 ppm were being denitrified without any long term perturbations to the throughput of the fermentor (which continued to operate in excess of a 10% dilution rate).

6. SEL BLEND COMPOSITION:

One of our foremost project goals was to determine the effect of changes in blend composition on the denitrification capacity of the mixed culture. With the assistance of Grant Gelatly and the R&D Pilot Plant crew, 100% Burley, Bright and Oriental lamina CELs were produced, together with 100% Bright and Burley stem CELs. Each was then diluted to a 7-8% solids level, fortified with KNO_3 to ≈ 2500 ppms, and introduced into actively-denitrifying fermentors. As mentioned previously, the 100% stem SELs (Burley and Bright) ran well, while the 100% lamina SELs (Burley, Bright and Oriental) did not. In fact, the successive addition of the 100% lamina SELs to the actively denitrifying fermentors resulted in a more or less immediate deterioration of the dilution rate from well above 10% to well below 10%. The mixed culture denitrification system does not appear to be able to handle 100% lamina SELs. Mixtures of these 100% lamina and stem SELs will be examined in the near future to determine end points for the maximum % lamina content in SEL that this microbial system can run. Such experiments as these would be useful in determining the impact of future blend compositions changes on the NOD system running at Park-500.

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7. PRE-ADJUSTMENT OF FEED SELs:

Although it has previously been demonstrated that this system requires no exogenous additives, recent experience has shown that a slight upward adjustment of the pH of our sterilized feed SELs generally promotes more consistent process operation and better throughput (all other factors being held equal). As a result, 1 1/2 to 3 mls of 30% Ammonium Hydroxide are currently being added to our sterilized SEL feeds to raise their post-autoclaved pH from about 5.2 to approximately 6.0. Alternative bases such as KOH and DAP have also been used in a similar manner with equal success, although the prolonged use of DAP-adjusted feeds did result in the build-up of large quantities of a brittle "scale" on the inside of the process fermentors necessitating frequent cleaning - thus, interruption of the processes.

Experiments conducted in the winter of 1982 using '82 blend composition Pk-500 SEL demonstrated that higher O-rates could usually be achieved by supplementing low NO_3^- feed SELs (1400-1800 ppm) with additional quantities of nitrate. Elevating such feeds to a ≈2500 ppm (+/- 300 ppm) level using KNO_3 resulted in higher and more consistent O-rates. However, we currently find that the mixed culture system runs equally as well on '83 blend Pk-500 SELs having "as-is" nitrate levels anywhere between ≈1400-2400 ppm. Higher nitrate levels (>3000 ppm) may or may not run better, but we now know this to be highly dependent upon the chemical quality of our SELs as metabolic substrates for the mixed culture (SELs with 3-5000 ppms NO_3^- have been run at dilution rates in excess of 10%).

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8. START-UP AND SHUT-DOWN PROCEDURES:

The specific details of our current operating process including start-up and shut-down procedures are given in our March 31, 1983 memo to

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Dr. J. Whitby, appended herein (Appendix A). Recent experiences with 10% - 30% inoculum "batch" start-ups have taken us a step further by demonstrating (to us) how critically-dependent such de novo batch start-ups are upon 1) the exact level of inoculum used (thus, number of viable microorganisms within), 2) it's past history with regard to age and "condition," and 3) the temperature of the start-up. It now appears that although "older" seed (>2-3 months old) will start up, (albeit, slowly), newer seed is usually more reliable in that it invariably starts up faster. Furthermore, independent of the relative quality of the stored seed, maximization of the percent (viable) inoculum used at initial start-up maximizes the chances of success. It is now evident that successful (reliable) short-term start-ups (<24 hrs) from refrigerated seed are highly dependent upon the presence of a critical minimal population of viable and "competent" microorganism(s) upon which the functionality of the entire microbial population depends under the unique environmental pressures of a low level inoculum (10-30%) batch start-up in chemically-variable SELs. We do not as yet fully understand the basis for these observations, but we do hope to be able to more fully elucidate these phenomena when more is known about the metabolisms and proportions of the individual participants within our mixed culture. Additionally, we now find cold (18°C) de novo batch start-ups to be generally more efficacious than those previously implemented at 47°C. Currently, refrigerated seed (stored at ≈4°C) is allowed to warm at room temperature to 14-18°C and is then added as a 30% inoculum to a sterile fermentor containing 7 ls of cooled (≈16-18°C) sterile SEL, pH 6.8 to 7.2, 1800-2200 ppm NO_3^-. The optimization of consistent and reliable de novo start-ups from inactive, stored seed still remains one of our chief goals. We are currently exploring alternative techniques and procedures for attaining this goal.

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9. PRODUCTION OF SEED FOR THE R&D PILOT PLANT START-UP:

In March 1983, start-up inocula (seed) was produced in the laboratory from Pilot Plant SEL, the composition of which was generally modeled after the 1982-83 Pk-500 SEL blend compositions. Approximately 80-100 liters of seed were collected aseptically in 2 l aliquots and stored at 2-4°C. Several samples of this seed have recently been used as 30% inocula to successfully start up fermentors in the laboratory.

10. CHARACTERIZATION OF THE MIXED CULTURE:

Experiments are currently under way in collaboration with Dr. Walter P. Hempfling, University of Rochester, NY, to elucidate the identities and capabilities of each of the microorganisms within the mixed culture. This information will be used to further optimize this process in the future.

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CONCLUSIONS AND SUMMARY:

1. The mixed culture of tobacco-identical microorganisms has been shown to be a stable and potent mediator of the thermotolerant, anaerobic dissimilatory denitrification of our Pk-500 SELs.

2. Industrially acceptable dilution rates in excess of 10% volumes/hr have been demonstrated over prolonged periods of operation in the semi-continuous mode over a wide range of SEL conditions including:
   a. Nitrate-N levels between 1400 and 3200 ppm.
   b. Feed Nitrite levels up to 800 ppm.
   c. Total solids levels to 14%, and
   d. Total alkaloids to 3.0%.

3. The reliability of process operation, temporary hold, shut-down and re-start (re-activation) procedures has been improved substantially as a result of an increased understanding of the impact of substrate (Nitrate) and environment (Temperature) upon the mixed culture.

4. We continue to investigate the phenomenological basis for both problematic and efficacious de novo start-ups from refrigerated seed. Progress has been made, within the limitations of our current understanding of this process, to improve start-up reliability by 1) increasing the initial inoculum used to 30%, and by 2) avoiding sharp thermal transitions (shock) at the time of inoculation by employing a cold, 13°C start-up procedure similar to our laboratory short-term operational shut-down and start-up procedures. Alternative procedures are also being investigated.

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5. Experiments on the impact of individual (100%) SEL blend components on NOD have demonstrated that 100% lamina SELs are not acceptable substrates for the curent mixed culture denitrification process, but that 100% stem SELs are.

6. Collaborative studies with a wide variety of personnel within PM R&D and Engineering have elucidated numerous process and operating parameters important to both our own understanding (and improvement) of our process, as well as to the design and engineering of the R&D Pilot Plant and Park-500 NOD facilities.

7. Certified seed has been made (and tested) for the Pilot Plant start-up in May, 1983.

8. Antibody to ATCC-1 has been made and tested. The resultant immunofluorescent tests demonstrate the presence of organisms similar in antigenicity to ATCC-1 in the mixed culture.

9. Evaluations of laboratory NOD product are currently under way to characterize their subjectives.

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ADDENDUM:

Although we are far from mastering an understanding of the intricacies of this microbially-mediated denitrification process; we have, with the help of an ever-growing community of collaborators, been able to substantially improve our appreciation for the strengths and weaknesses of our current NOD process. This has recently led (directly, or not) to advances in the reliability and consistency of our process. We are confident that continued studies in these areas will even further enhance our ability to understand and utilize this and future biotechnology processes to make commercially-successful products here at Philip Morris.

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ACKNOWLEDGMENTS AND PERSONNEL:

The progress made to date in our understanding of the laboratory-scaled NOO mixed culture system is largely due to the extensive efforts of each of the personel within Project 1904, many of whom worked unusually long hours over extended periods of time to keep our long-term semicontinuous fermentation experiments going. Special thanks are extended to Robert Dunn for his diligence and efficiency in helping schedule both personnel and experiments for the numerous interrelated and overlapping investigations that have been conducted over these past six months.

Project 1904 personnel would also like to acknowledge their appreciation for the extensive collaborative efforts of a variety of other researchers and facilities within PM R&D, including: Duane Watson, et al., (GC); Betty Handy and Bill Harvey, et al., (Analytical); Grant Gellatly, et al., (Pilot Plant CELs); Bob McEuen and Edie Taylor (Immunofluorescence); and Ray Harding and other Pk-500 personnel (SELs).

We would also like to thank our PM management and scientists for their continued support (incl., William A. Perone, Jerry Whidby, Cliff Lilly and Ved Malik), and our consultants for their valuable suggestions and guidance, Walter P. Hempfling (University of Rochester, NY), and William Stark (PEDCo).

Project 1904 Personnel are: J. Banyasz[1], P. Kusel, R. Dunn, D. Chadick, S. Tennet, P. Oglesby, E. Bravo, G. Fortner[2], P. Boone, G. Nixon[1], A. Conyers, P. Finn, M. Varnier and I. L. Uydess.

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1. Currently in other projects.

2. No longer with PM R&D.

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APPENDIX A

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PHILIP MORRIS U. S. A.

INTER-OFFICE CORRESPONDENCE

RICHMOND, VIRGINIA

To: J. F. Whidby Date: March 31, 1983

From: I. L. Wycass

Subject: Operational and Process Parameters of the Mixed Culture Denitrification System

I. DE NOVO START-UP FROM STORED, REFRIGERATED SEED

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Definition of:

A. SEED: Start-up inoculum consists of 1-2 liters of refrigerated product from previous successful denitrification run(s) using Park 500 SEL as feed. Product from said run (cycle) is collected in a sterilized polypropylene (polyethylene) 2-liter, screw cap storage jar and placed immediately into the cold room after being labeled with the following information:

- I.D. Number of Fermentor Vessel of Origin
- Denitrification Run (Cycle Number) From Which Seed Was Collected
- Date and Time of Collection
- ppm's NO_3 and NO_2 in Product
- pH of Product
- Presence or Absence of Butyric Acid (Normally Never Present in Product)

Additional data of import regarding the overall "quality" of the product (and potential inoculum) that is recorded in lab notebook(s):

- Cycle Time of That (and Every) Run
- Extent of NO_3 and NO_2 Accumulation During Run (Cycle Prophile)
- Initial and Final pH of Run
- Nitrate Level of Feed Used in That Run
- Cell Number in Fermentor (Thus Product)
- HPLC Organic Acids Data (Acetic, Propionic, Butyric, others)
- Off-Gas Profiles Typical of Collection Period

Normally, only product from successful runs is collected for storage as seed. This means:

- That the cycle time at the time of collection of the product (seed) was at least 0.1 "D" or better (typically, a dilution rate of 15%).
- That NO_3 and NO_2 are N.D. in the product (NO_2 is typically 50-100 ppm at the time of collection but goes to zero immediately afterwards).
- That there is no butyric acid in the product (or in the fermentor producing the product).
- That the phenomenology of NO_3 and NO_2 removal during that run was "normal"--e.g., that there were no unusual or significantly high

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accumulations of either NO_3 or NO_2 during the run. (Otherwise, this would be indicative of a problem in removing NO_3 and/or NO_2 during that cycle).

. That the product is from the denitrification of a "standard" SEL--not a specialized SEL formulation being used for experimentation.

. That the nitrate in the feed SEL being denitrified at the time of collection of the product (seed) was within the normal recommended range (approximately 2200-2800 ppm NO_3).

. That the feed SEL used during the collection of that seed had no "off" characteristics--such as: 1) the presence of "out-of-spec" quantities or organic acids (proprionic or, especially, butyric), 2) >100 ppm NO_2, and/or 3) an unusually high initial pH that is not known to be due to a change in blend composition, but rather, to be indicative of prior microbial activity in the feed.

3. FEED: Acceptable feedstock for mixed culture denitrification has been defined as SEL product from the PM Park 500 facility which has the following "as-is" characteristics:

1200 to 3200 ppm's NO_3 (depending upon blend composition and line of origin).

Butyric Acid - None (unless this is known to be due to blend components used and not to prior microbial activity).

Propionic Acid - None (unless this is also known to be due to blend components used and not to prior microbial activity).

. Acetic Acid no higher than 3-4,000 ppm.

. "As-is" pH of 5.3 to 5.8. (This depends upon SEL blend composition and may vary slightly due to the presence or absence of higher or lower proportions of Burley vs. Bright, stem vs. lamina, etc.) *However, unusually high pH can also be indicative of prior microbial activity, but this is usually accompanied by unexpectedly low NO_3 levels and high NO_2 levels and may also be accompanied by higher than normal concentrations of proprionic and/or butyric acids.

. Post-sterilization (post-autoclaving) pH of 4.9 to 5.4. Again, this is somewhat dependent upon the original "as-is" pH and can be blend dependent. However, unusually low pH's (lower than pH 4.9) have, in the past, been indicative of possible off-spec.-chemistry SEL (high concentration of bright lamina?) which are more difficult to run and often result in longer cycle times, lower "O" rates, and poor operational consistency.

. Soluble Solids: nominally 6-10% (5-15% have been run).

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. Insoluble Solids: (We have not tested this yet.)

. Total Solids: nominally 6-10%.

. Total Alkaloids: nominally 0.1-0.3%.

In-house Adjustment of Feed:

Purpose: In order to increase the efficiency and optimize the consistency of the mixed culture denitrification process, the following guidelines have been adopted:

. Increase NO_3 (if necessary) to 2500 ppm +/- 300 ppm by the addition of KNO_3 prior to sterilization.

. pH adjustment of the post-sterilized feed SEL to between pH 5.8 and 6.0 by the addition of 30% NH_4OH.

C. DE NOVO START-UP: When a complete restart of process is required necessitating the utilization of a small quantity of stored, refrigerated seed which must be amplified by serial (1:10) increases in volume.

1. Feed Start-up:

. One or two liters of stored inoculum is transferred aseptically into a sterile, 14-liter fermentor vessel already on a fermentor mainframe and hooked up to a sterile, preadjusted feed (approximately 2500 ppm NO_3, pH 5.8-6.0). Alternatively, the seed could be added to a vessel that already contained 1-2 equivalent volumes of sterile, preadjusted feed. In this instance, this initial volume of feed may have to be denitrified before feeding of additional SEL is begun.

. Feeding is then initiated at approximately 0.1 volumes/hr (10% volume per hour) using 47°C preadjusted feed.

. NO_3, NO_2 and pH are monitored periodically throughout the fill (usually at each 0.1 volumes of addition--e.g. at each 1 liter of addition of feed to a final working volume of 10 liters in the 14-liter fermentor). The fermentor itself is set to operate at 47°C.

. If, during the initial adjustment period (of increasing temperatures and the addition of fresh feed) NO_3 and/or NO_2 levels build to greater than 100-150 ppm NO_3 and/or >300-400 ppm NO_2, feeding is temporarily discontinued until NO_3 goes to N.D. and NO_2 decreases to ≤100 ppm. Feeding is then resumed at 0.1 "D".

. Monitoring of NO_3, NO_2 and pH should be continued (at each addition of 1 liter during fill) to verify efficacy of start-up.

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- When final working volume has been reached (10 liters), and the reactor is verified to be N.D. NO_3 and <100 ppm NO_2, one-half (1/2) of that total volume (e.g. 5 liters) is pumped off rapidly until a 5-liter heel remains. Discharge of product is then discontinued, and feeding (at 0.1, 0.15, or whatever feed rate is required by system) is reinitiated until the final 10-liter volume is attained again.

- NO_3 and NO_2 (as well as pH) should be monitored each 0.1 volumes during fill to again verify the operational status of the system (that it is running at ≤100 ppm NO_3 and ≤250 ppm NO_2 during fill--otherwise, an adjustment in feed rate may be required).

- If NO_3 and/or NO_2 are not approximately N.D. (N.D. NO_3 and ≤100 ppm NO_2) at end of fill, a variable post-fill "finishing" period may be required. This can be normal, and often depends upon (and reflects) the "quality" of the feed SEL and operational status of the mixed culture. However, when the process is running at a "D" rate of 0.1 or better (0.15, etc.), such a finishing period should not exceed 10-30% of the fill time (depending upon the length of the fill time).

- Typically, in a 5-liter heel system, the fill time should be approximately 3-3.5 hours, and denitrification should be completed either when the fermentor is full, or within 30 minutes to one hour thereafter.

2. STRAIGHT BATCH START-UP: Alternatively, the NOD process can also be started from stored, refrigerated seed using a 1:10 batch start-up.

- One liter of refrigerated seed is transfered aseptically into a sterile, agitated 14-liter fermentor containing 9 liters of preadjusted SEL feed held at 47°C.

- NO_3, NO_2 and pH are then monitored at 1-2 hour intervals until denitrification of the initial 10-liter volume of feed (feed plus seed) is verified.

- Fifty percent of that fermentor volume is then discharged rapidly and the addition of fresh feed begun at approximately 0.1 volumes (10%) per hour until that fermentor is full once more.

- NO_3, NO_2 and pH are monitored at each 0.1 volumes of addition (in this case, at each one liter of addition) to ascertain the operational status of that fill.

- If denitrification is not completed by the end of the fill interval, a variable post-fill finishing period may be required. NO_3, NO_2 and pH samples are taken as needed during this interval until denitrification is complete. However, as stated before, this period should not exceed 20-30% of the fill time, depending

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upon 1) the actual duration of that fill time, 2) the feed NO_3 concentration, and 3) the previous "O" rate of that system).

. The interval required for the complete denitrification of the initial 10-liter volume of feed is typically between 15 and 30 hours, depending upon the "quality" of both the feed and the seed.

II. PROCESS OPERATION: (Assuming a 50% Heel)

A. Shutdown: In the event of a planned shutdown for greater than 12 hours (typically, for one day to two weeks), during which time 25% or more of the maximum fermentor capacity would be held and maintained at 4°C (in the fermentor) to be used as inoculum for a subsequent run:

. On the last planned cycle, slow fill the first half of the fill volume at the nominal fill rate, then batch in the last half of the fill volume. Stop heating; cool to 4°C. Stop agitation; maintain at approximately 4°C.

B. Start-up: (from refrigerated full or partially full fermentor containing "held" product):

. Discontinue cooling, resume agitation and when fully mixed, discharge 50% of contents. Begin heating to 30°C. When fermentor temperature reaches 20-25°C, resume feeding (initially at about 10% per hour) until full. When NO_3 and NO_2 are zero, discharge product and resume normal fill cycle (normal operation).

To Be Monitored While Feeding During Initial Start-up Cycle): NO_3 and NO_2 levels in fermentor must be checked--usually as each additional 10% of volume is added to determine status of the mixed culture (e.g., are NO_3 and NO_2 being consumed normally, or not?). Usually, bacteria will become active (with respect to their ability to remove NO_3 and NO_2) above 25°C. If they are "healthy" (if previous shutdown did not perturb culture and, if current SEL is acceptable), the mixed culture will consume incoming NO_3 and NO_2 during warm-up. Organic acids (acetic, proprionic and butyric) must also be monitored to verify status of "held" (refrigerated) culture. Normally, some proprionic acid (200-300 ppm) may be found to have accumulated during cool-down and/or storage. This could be due to metabolic changes resulting from the progressive temperature shifts experienced by the culture, or, because of alternative metabolisms be expressed if NO_3 and NO_2 are depleted during cool-down before the mixed culture becomes metabolically inactive. No butyric acid should be present in the start-up mixture. Microscopic cell counts and viable plating should also be done to verify cell number.

C. TEMPORARY HOLDS (SCHEDULED, OR OTHERWISE):

When a temporary disruption in the operational status of the NOD facility precludes either A) the discharge of a finished volume of product, or, B) refeeding of the Saved Heel (e.g., starting a new cycle), the following procedure can be employed:

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- "X" volumes of a concentrated, sterile solution of KNO_3 may be added (batch or fed) to the full or half-full fermentor containing the denitrified product so as to raise the NO_3 level in that product to between 1000 and 2000 ppm.

- NO_3, NO_2 and pH are then monitored at 1/2 to 1 hour intervals until all of the added NO_3 (and resultant NO_2) is removed. The length of time that this denitrification cycle would take is predictable, and would be related to 1) the prior NO_3-removing capacity (history) of that fermentor, 2) the amount of NO_3 added, 3) the extent of the microbial population, and 4) the "quality" of that fixed volume of SEL (with particular regard to the persistence of excess substrates remaining in that volume of previously denitrified SEL).

- Once this additional NO_3 has been removed, normal process operation should be resumed.

- The efficacy of this short-term (one cycle) holding procedure is limited and highly dependent upon the substrate quality of the original feed SEL.

D. NOMINAL PROCESS OPERATION:

Once the system is up and running, a "D" rate of 0.1 hours^{-1} should be attained within 2-3 cycles after start-up (if no complications arise due to [illegible] problems experienced prior to or during shutdown or start-up). If all is well during shutdown and start-up, the initial cycle of start-up should take no longer than 8-12 hours, assuming a 50% refrigerated heel, 10% feed rate, and the normal delay in microbial activity experienced as a result of the temperature shift from 4°C to 47°C. Each subsequent cycle should then be 50 to 75% the time of the previous cycle until the nominal 0.1 "D" rate of 5 hours is reached (again, this is normally attained within 3-4 cycles, including the initial, cold, start-up cycle). Worst case thus far, 28 hours to attain ≥0.1 "D"; and best case, 7 hours to attain ≥0.1 "D". It is normally 8 to 12 hours.

E. NOMINAL PROCESS MONITORING: When working with a new (as of yet, unfamiliar) system.

- NO_3 and NO_2 should be checked at each 10% volume level increase during fill and at required intervals afterwards if product is not completely denitrified by the time of completion of fill.

- pH should be monitored during fill and especially during final interval of denitrification (nitrite removal) to verify "normal" phenomenology of cycle (e.g., that the pH increases as NO_2 goes to N.D., and also, that the final pH at the time of completion of the run is about the same as that of previous runs). However, this is also somewhat dependent upon normal day-to-day variations in the overall quality of the Park 500 SEL with regard to: 1) chemistry of the incoming feed (blend and/or other

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compositional changes affecting buffering capacity, etc., and 2) initial NO_3 level in the feed (the higher the feed SEL NO_3 level, the higher the resultant finishing pH).

Other parameters to be monitored:

- Cell Number: once per shift or whenever a change occurs.
- Organic Acids (HPLC): once per shift or whenever a change occurs.
- Off-Gases (GC): periodically or whenever a change occurs.
- Colony Morphology (Plating): interval varies (daily).

F. PROCESS MONITORING: (Working with a known system.)

Once some familiarity (experience) with the system has been acquired:

- NO_3 and NO_2 should be checked at end of the fill cycle and, if required, subsequently at the completion of denitrification.
- pH at end of fill cycle (and, if required, subsequently at completion of denitrification).
- Cell Number: once per shift and whenever a change occurs.
- Acids: once per shift for both the feed and fermentor(s) or whenever a change occurs.

III. WHAT WE KNOW ABOUT "GOOD" AND "BAD" CYCLES:

A. Characteristics of "Good" Cycles:

- "D" rate $\geq$ 0.1 hrs^{-1} (typically 0.15 hrs^{-1} or 15%).
- NO_3 never accumulates to more than 50-100 ppm during fill (usually by mid fill and $\leq$50-100 to N.D. by end of fill).
- NO_2 does not accumulate to more than 350 ppm during fill (typically 50-250 ppm by end of fill) and goes to N.D. within 30-45 minutes after fill.
- pH decreases slightly during initial period of fill (due to addition of acidic SEL feed), but increases during last half of fill (and/or finishing) due to the removal of NO_2.
- pH at end of fill approximately same as "finishing pH" of previous good cycles.
- No butyric acid produced.
- No propionic acid produced.

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. Cell number $\geq$ $1x10^9$ cells/ml (typically 2 to $4x10^9$ cells/ml).

. N_2 and N_2O major off-gas products (as opposed to CO_2).

3. Characteristics of a Problematic Cycle:

. "D" rate <0.1 hrs^{-1} (or an abrupt decrease in "D" rate from previous cycle - e.g., $\geq$50% of that previous "D").

. NO_3 accumulates to more than 150 ppm during fill.

. NO_3 present (to over 150 ppm) after completion of fill.

. NO_2 $\geq$350 ppm during fill.

. NO_2 increases to greater than 300 after completion of fill (as the residual NO_3 goes to NO_2).

. Time to finish after fill is more than 1/2 of total fill cycle.

. pH remains low throughout fill.

. pH at end of fill and/or end of finishing is significantly lower than "normal" finishing pH of previous runs unless this is known to be due to a blend or feed nitrate level change.

. Some butyric acid produced (if present, indicative of problem possibly related to "quality" of feed).

. Propionic (although this is variable, and 200-300 ppm's are not uncommon during periods of successive, slow runs).

. Cell number of $<1x10^9$ (or abrupt decrease in cell number). Cell number - (total cell count or viable count) may not decrease if slow cycle time is related to a transitory respiratory or metabolic problem that manifests itself in slow NO_3 and NO_2 removal only. However, slow cycle times accompanied by a significant decrease in cell number is indicative of a much more significant microbiological problem affecting cell growth.

. CO_2 major off-gas (proportion of CO_2 to N_2+N_2O increase).

/lvb

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INTER-OFFICE CORRESPONDENCE

RICHMOND, VIRGINIA

APR 29 1983

J. F. HARVEY

To: Distribution

Date: April 27, 1983

From: K. H. Cho

Subject: PROJECT CANCELLATION - N.O.D. PLANT - PARK 500
PROJECT NUMBER BC26-0002

This is to notification that N.O.D. Plant - Park 500 project (Project Number BC26-0002) has been cancelled effective April 26, 1983.

Pedco Inc. has been notified to stop all activities for this project. Project closing work with Pedco Inc. is in process.

All project documents will be filed in Central File at the Operations Center. Each member of the project team may keep his own file in his own way. If you prefer to turn over to central file, please hand me the files by May 5, 1983.

Although it was a short time, I appreciate your cooperation and it was a pleasure working with you.

K. H. Cho

/pa

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PHILIP MORRIS U. S. A.
INTER-OFFICE CORRESPONDENCE
RICHMOND, VIRGINIA

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MAR 24 1982
PHILIP MORRIS

Please file 2000 1 Copy

To: Mr. H. G. Burnley, Jr. Date: March 19, 1982

From: J. C. Gomes

Subject: STATUS REPORT - M.O.D. PROJECT
PROJECT NUMBER BC26-0001

Attached for your information and review is a report on the status of the M.O.D. project. It consists of:

I. OBJECTIVE
II. CONCLUSIONS
III. PLANS
IV. DISCUSSION

J. C. Gomes

/bwb

Attachment

cc: Mr. A. S. Church
Mr. J. F. Whidby
Mr. J. A. Williams
Central File BC26-0001

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SUBJECT: N.O.O. PROJECT STATUS

I. OBJECTIVE

Present an overview status of the N.O.O. project for management review.

II. CONCLUSIONS

1. Denitration by bacterial fermentation has been achieved in the pilot plant, both by fed-batch and continuous mode operation.

2. Subjective indications of product quality and acceptability are favorable and official evaluations are in progress. However, quality and acceptability of product denitration by bacterial fermentation has not yet been approved.

3. Consistent denitration rate results have not yet been achieved in the pilot facility. This is primarily attributed to variations in feedstock composition (i.e., solids, nitrates, nutrients, inhibitors, etc.) and excessive levels of contaminating organisms.

4. Successful process startup has been repeated in the lab several times, but only once in the pilot plant. This was done using pH adjustment and inoculum grown and harvested in sterile SEL.

5. Sterilization of the entire process feedstream will be required to control contaminants which produce unacceptable levels of organic acids. Primarily butyric acid.

6. Excessive levels of organic acids are unacceptable because of the odor and taste they introduce to the product. Subjective testing is in progress on sheet runs made with SEL denitrated in the pilot plant during February 1982, to determine acceptable acid levels.

7. Pilot plant modifications will be required to sterilize and maintain levels of contamination acceptable to this process.

8. The known process conditions at this time are pH (between 7 and 8.5) and temperature (50°C ± 2).

9. The nutritional requirements and growth inhibitors for the desirable organism are not yet completely known.

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10. The impact of variations in feedstock composition on the process are not yet known.

11. The process configuration for this bacterial fermentation process will require four main steps.

    a. pre-sterilization
    b. seed line
    c. process fermentation
    d. post-pasteurization or sterilization

12. Conceptual control techniques have been successfully developed and operated in the pilot facility.

    - nitrite monitor
    - pH monitor and control
    - integral P.C. controlled process

13. Major equipment requirements have been established. However, evaluation of the specific items has not yet begun.

14. Work on analytical requirements and procedures are underway. However, all of these are not yet available.

15. It is the opinion of our consultant (R. Hatch) that approximately 60% of the required data for process definition has been obtained. The remaining 40% consists primarily of fine details, process optimization and successful repitition of results and confirming data.

III. PLANS

1. Make necessary pilot plant modifications to be able to control contaminants.

2. Define the impact of variations in feedstock (nitrate and % solids) on the denitration process using DNCEL and diluting to desired concentrations.

3. Verify the reliability of the new startup procedure, using pH adjustment and inoculum grown and harvested in sterile SEL.

4. Define nutritional requirements for growth and growth inhibitors for the desirable organism. A future study will also be conducted to define the presence and variations of both nutrients and inhibitors in the SEL feedstock.

5. Identify and evaluate required process equipment.

6. Produce 3000 to 4000 pounds of RL sheet denitrified by the bacterial fermentation process to conduct product evaluations and mail out tests.

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7. Define the effluent gas composition and related environmental impact and control requirements.

8. Once acceptable results are achieved from the pilot plant, a sheet run will be made at Park 500 with SEL denitrated by bacterial fermentation. This is to better determine the product quality and acceptability.

9. A consultant firm with primary expertise in the development of bacterial fermentation processes, will be identified and brought in to work with PM personnel on the development, scale up, and engineering of the N.O.B. process.

DISCUSSION

Until late Fall 1981, the denitration pilot plant at R&D was using SEL from Park 500 and achieving good levels of denitration. The process was only operated in the feed-batch mode and batch times were in the range of 3 to 4 hours. However, the pilot facility did not provide much in the area of controls and flexibility. Also, the equipment configuration was not desirable, because it did not provide the ability to run the process like a fermentation plant should be run.

For these reasons it was decided that a new pilot facility was necessary. The new facility would provide more automation, controls and flexibility, and would be in a configuration more like a bacterial fermentation production facility. This would consist of four (4) steps:

A. pre-pasteurization
B. seed line
C. fermentation process
D. post-pasteurization

From November 16, 1981 through January 18, 1982 this new pilot facility was designed and built.

Once the equipment startup problems were resolved, several attempts were made to startup the seed line process with no success. The procedure used for this startup was the same that had been used for previous startups, but this time it would not work.

After approximately six unsuccessful attempts over a three week period, a difference in pH response was observed. It was noticed that each time the culture was transferred from the synthetic growth media to SEL, nitrate would start being converted to nitrite, pH would drop below six (6) and a failure resulted. Figure 1 is the pH curve during each unsuccessful startup attempt.

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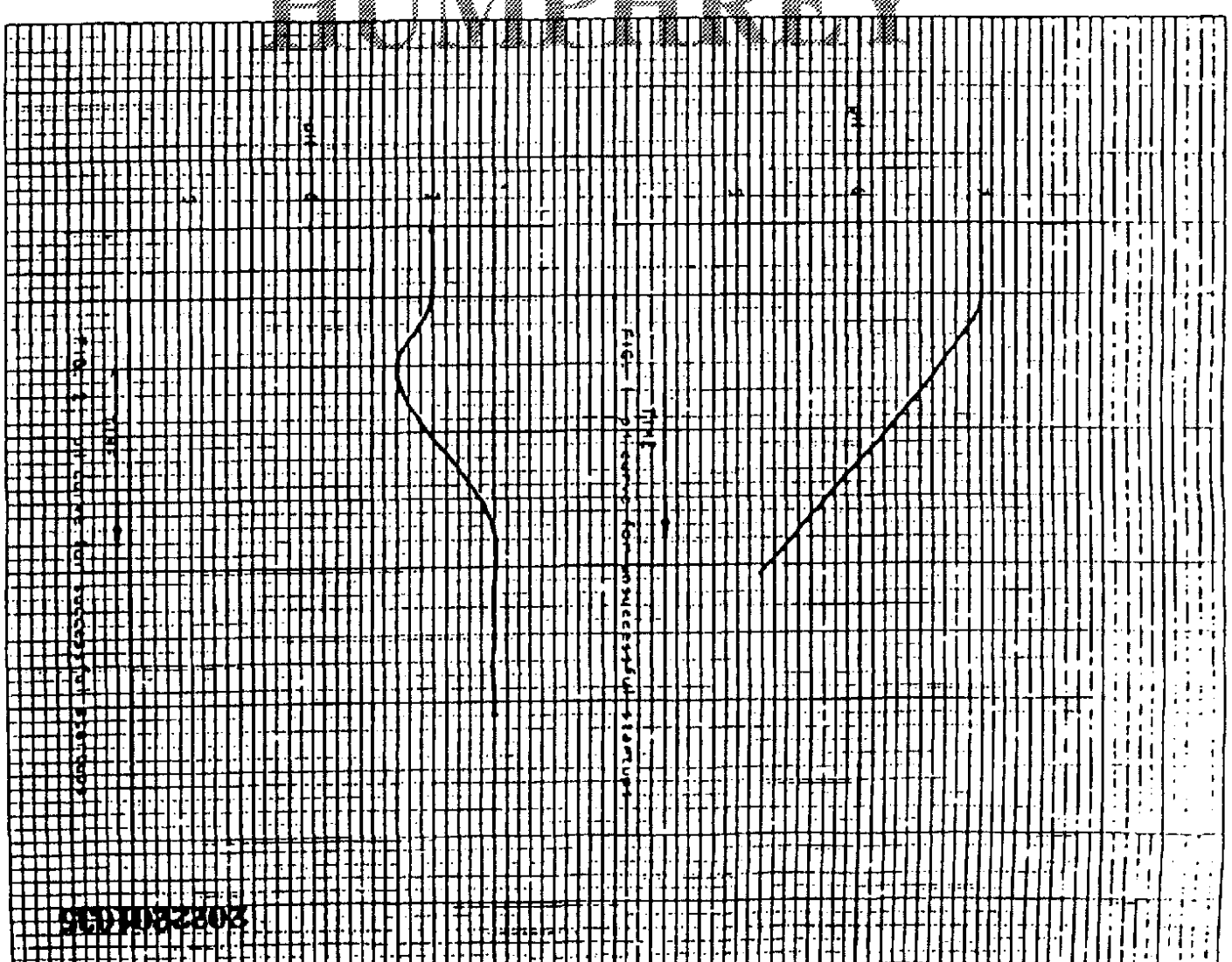

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When Figure 1 was compared with Figure 2, the pH curve for successful startup, instead of a steady drop in pH, a slight dip followed by a rise to a point of constant pH was observed. This was followed by the consumption of nitrite and successful denitration.

For the next startup the pH was adjusted so that it would not drop below 6.5. This resulted in a successfuly startup.

At this time, the exact cause of the problem is not known. What is known is that if the organism is grown and harvested in a media other than SEL they will adapt their metabolism for that media. When transferred to an SEL as media, these organisms will require some time to adapt their metabolism.

A new startup procedure is now being used. To reduce the time required to adapt the organism to a new media, the organism to be used for inoculum is grown and harvested in sterile SEL, concentrated and put into frozen storage for use when a startup is necessary. The new procedure also consists of initial pH adjustment of the startup media. This procedure has been successfully repeated several times in the lab but only once in the pilot plant. The previous best startup was 18 hours and the first time with the new procedure in the pilot plant it took 6 hours to startup.

Until the startup of the new pilot facility, almost all the work had been done with a mixed culture, consisting of many types of organisms. Since then, only pure cultures (single organism culture) have been used and significant efforts directed at maintaining the desired single organism predominant in the seed line.

Consistent dilution rates (rates of denitration) have not been achieved. This is primarily attributed to variations in SEL feedstock and contaminating organisms. Even though no data exists yet to substantiate that feedstock variations (nitrate and % solids) affect denitration, some indications are that it does. A test program to test this using ONCEL (4200 ppm NO_3N and 46% solid) and diluting it to the desirable concentrations is planned.

The redesigned pilot facility has added more sophistication in controls and data acquisition. Also the R&D analytical support on the project has been increased to provide more detailed up-to-date data (organic acids, nitrate, nitrite, pH, etc.)

With this added flexibility R&D has been able to determine that contaminating organisms exist in the process and these produce high levels of organic acids (acetic, butyric, and proprionic). The presence of the contaminants appears to be tolerable up to a certain level, but in excess can significantly upset and even wipe out the process.

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Significant amounts of data exist that the presence of contaminating organisms not only impact significantly on the rate of denitration but also produce extremely high levels of organic acids, primarily butyric acid, which introduces an extremely objectionable odor and taste to the product. Subjective testing is currently underway on sheet made with SEL denitrated in the pilot plant during the month of February. This testing will provide some data on what levels of these organic acids will be acceptable.

For this reason it is now unanimously accepted that sterilization of the entire SEL feedstock to the process will be required as well as means to maintain contaminants at or below critical level. The new pilot plant was not designed with the capacity to sterilize the entire SEL feedstream, only approximately 10%. This was because pasteurization of the rest of the feedstream was thought to be adequate. It was also felt that this process fermentor would not require provisions to keep airborne contaminants and others out because the desired organism would be kept predominant with a constant feed from the seed line. Therefore, the process fermentor was specified as an open vessel and all associated equipment designed with maintenance of sterility not considered.

From the data now available on contaminants, organic acids, sterilization and pasteurization will be necessary to sterilize the entire feedstream to minimize the introduction of contaminant organisms throughout the process. To do this, several modifications will be required to the pilot plant.

Approximately three months ago, R&D brought in a micro-biologist (Dr. Peter Kusel). Dr. Kusel has identified the need for the completion of a significant amount of work to better define the requirements of this particular bacterial fermentation process.

In order for the organism to grow, it needs nutrients which are for the most part in SEL. We know this because often the organism grows at tremendous rates in SEL. What is now required for better control of the process is to determine what the organism needs to grow and then determine the presence and variation in levels of these nutrients in the SEL feedstock. In conjunction with this, there are also growth inhibitors that need to be identified and their presence in SEL established. Plans to carry out this work are underway.

Many of the major equipment requirements have been identified and in several instances options exist. However, evaluation of specific equipment items has not yet begun.

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Currently two consultants with expertise in bacterial fermentation processes and technology are being used. Dr. Harry Wang, Professor of Chemical Engineering at the University of Michigan, and Dr. Randy Hatch, Associate Professor of Biochemical Engineering at the University of Maryland. Both consultants are very familiar with this process and the specific work done. After receiving the latest data, they are of the opinion that at least 60% of the work required to define a process has been completed. The remaining work consists primarily of working out the fine details, process optimization, equipment selection and evaluation and successfuly repetition of the results and confirming data.

Fermentation technology (scale up, design and engineering) is a new field for P.M. and as such, in house expertise is non existent. Plans are underway to get some engineering personnel trained on the basics of Fermentation Technology this Summer at M.I.T. However, this is only and introduction to basics.

Since PM Engineering's expertise is lacking significantly in fermentation technology, and since the R&D expertise in the same field is also limited, a consultant firm is being sought that can fill the gap.

The chosen consulting firm will work with both R&D and Engineering to insure the necessary data is generated in the pilot facility, properly analyzed and scaled up.

As of this writing, a significant effort has been put forth on this task and a list of 16 potential candidates has been narrowed down to four.

The current project priorities are:

A. <u>In the pilot plant</u>

1. Make required modifications to be able to control contaminant.
2. Evaluate impact of NO_3N and solids concentration on the process.
3. Successfully demonstrate ability to reliably startup.

B. <u>In the lab</u>

1. Define the nutrients and inhibitors for the growth of the organism.
2. Develop a reliable startup procedure.
3. Complete the study on the chemistry of SEL.
4. Identify effluent gas composition of this process.

The organization, scheduling and priority setting for this work is the subject of bi-weekly meetings of the entire project team. During this meeting the process definition and development items list is reviewed, updated and priorities set.

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United States Patent Office 3,420,241 Patented Jan. 7, 1969

3,420,241
METHOD OF PREPARING A RECONSTITUTED TOBACCO SHEET EMPLOYING A PECTIN ADHESIVE

John D. Hind and Robert B. Seligman, Richmond, Va., assignors to Philip Morris Incorporated, New York, N.Y., a corporation of Virginia

No Drawing. Application June 16, 1966, Ser. No. 557,903, which is a continuation-in-part of application Ser. No. 336,009, Jan. 6, 1964. Divided and this application Apr. 28, 1967, Ser. No. 647,277
U.S. Cl. 131—140 1 Claim
Int. Cl. A24b 1/14

ABSTRACT OF THE DISCLOSURE

This disclosure relates to a process for producing a binder composition for use in the manufacture of reconstituted tobacco. The binder is made from tobacco plant parts and involves the use of the naturally occurring tobacco pectins, which are obtained by a process in which ammonium orthophosphate is employed to treat the tobacco plant parts. The treatment involves the destruction of the alkaline earth metal cross-links of the tobacco pectins, the release of the resulting tobacco pectins by a washing step, and the depositing of the released tobacco pectins on the treated plant parts.

This application is a division of application Ser. No. 557,903, which was filed on June 16, 1966 now Patent No. 3,253,597, and which, in turn, is a continuation-in-part of application Ser. No. 336,009, which was filed on Jan. 6, 1964, now abandoned and which, in turn, is a continuation-in-part of application, Ser. No. 240,130 filed Nov. 26, 1962, now forfeited and application, Ser. No. 189,995 filed April 16, 1962, now abandoned.

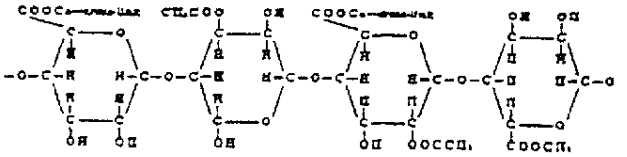
This invention relates generally to a method for the production of a reconstituted tobacco composition in which tobacco pectins within the tobacco itself serve as the binder.

During the preparation and processing of tobacco products, including aging, blending, sheet forming, cutting, drying, cooling, conditioning, shaping and packaging, considerable amounts of tobacco fines and tobacco dusts are produced. It is known that such tobacco fines and dust can be combined with a binder to form a coherent sheet, which resembles leaf tobacco and which is commonly referred to as reconstituted tobacco. One method for making reconstituted tobacco of this general character is disclosed in United States Patent No. 2,734,510, wherein the tobacco fines and dust are applied to a binder made of carboxymethyl cellulose carboxymethyl hydroxethyl cellulose or a suitable salt thereof. The binder, in such compositions, ranges from about 5% to about 50% of the weight of the tobacco employed. United States Patent No. 2,708,175, describes a binder for reconstituted tobacco which consists of a plant gum, principally of galactomannan. United States Patent 2,592,554 to Frankenburg describes, as binders for reconstituted tobacco, various water-soluble polysaccharides, such as alginic and pectinic acids and their sodium and potassium salts derived from plants other than tobacco; for example derived from citrus fruits. However, the addition of cellulosic binders further increases the amount of cellulosic material in the product and tends to create an acrid and bitter smoke, when the product is used to make cigarettes. The natural hydrophilic colloid gums such as guar gum, locust bean gum, algin and other commonly used materials, such as Irish moss, have additional disadvantages. These materials contain proteins and other materials not found in tobacco which add distinctive flavors of their own to tobacco products during smoking. Thus, Frankenburg, in describing the use of various water-soluble polysaccharides derived from plants other than tobacco, teaches that care should be exercised that they must be in a state of refinement. Frankenburg teaches that these materials should be free of extraneous matter containing compounds of nitrogen, particularly proteins, and compounds of sulfur, phosphorus and the halogens; i.e., compounds giving undesirable products of combustion or dry distillation. Such refining is often a very tedious and difficult operation.

The present invention makes possible the production of improved reconstituted tobacco by a method which is simpler and more effective than the methods previously employed. The present method does not require refining of the binder and it, therefore, more easily and efficiently employed than other methods for making binders and for making reconstituted tobacco. The reconstituted tobacco which is obtained in accordance with the present invention need not contain any additional cellulose or proteinaceous material which is foreign to tobacco, since the binder which is employed may be derived solely from tobacco, and may be produced in such a manner that it contains no materials other than those which naturally occur in tobacco. Thus, reconstituted tobacco produced in accordance with the invention, can be so formulated as to be similar in physical properties and chemical composition to natural tobacco.

The term pectic substances[1] will mean those substances which are found in many plant products, and which consist essentially of partially methylated galacturonic acids joined in long chains.

The pectic substances found in tobacco plants contain acetyl groups and differ considerably from commercially available pectins found in other plants, including sugar beet pectins and citrus and fruit pectins. Tobacco protopectins are uniquely insoluble in hot water as compared with protopectins from many other sources and comprise mainly water-insoluble pectins (protopectins) consisting of the calcium and magnesium salts of partially esterified



and slightly acetylated polymers of galacturonic acid. The divalent calcium and/or magnesium atoms act as cross-links between acid chains, thus making the polymers water-insoluble. As an illustration, the structure of the calcium salt of a polymer of galacturonic acid can be represented as above.

[1] Note: Unless otherwise specified, the term "pectins" will, for convenience hereinafter be employed interchangeably with the term "pectic substances."

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Although pectins have long been known as constituents of plant tissue, it has been found extremely difficult to separate pectins from the remainder of plant components and to obtain them as homogeneous compositions. The recovery of pectins from tobacco is even more difficult than the recovery of pectins from other plants.

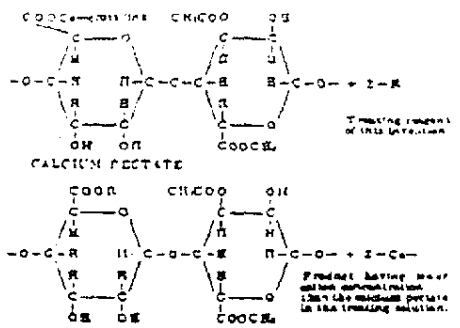
In accordance with the present invention, tobacco parts are bonded together by tobacco pectins which are especially prepared by a novel process which yields these pectins in a form in which they can be employed as binder materials. Our process for preparing tobacco pectins comprises first reacting tobacco parts, preferably in a form in which they present a large surface area, with an aqueous solution of a non-toxic reagent which is capable of reacting with and destroying the calcium and magnesium cross-links in the pectinaceous substances which naturally occur in tobacco. After the calcium and magnesium cross-links are destroyed, the tobacco pectins are liberated and made available for use as a binder. The tobacco pectins are then dissolved or dispersed in solution, or are at least sufficiently released from the interstices of the tobacco mass so that they form a coating on the surface thereof. Tobacco pectins which are dissolved or dispersed in the treating solution are thereafter precipitated or deposited from the solution so that they become available for use as a binder material. In this way, the tobacco parts can be bonded together by a binder material which is made of ingredients that are closely related to the naturally occurring ingredients of tobacco. The bonding can be accomplished without need for the purification of the tobacco pectins, inasmuch as any materials which are present are closely related to the materials which are normally present in tobacco and, thus, do not add any undesired qualities to the tobacco.

The tobacco parts which can be employed in the present invention comprise tobacco leaves, stems and stalks, and mixtures of these, whether in sheet, flake, particulate or other form. Preferably, the parts are ground, cut or otherwise prepared in a form which presents a large surface area. The midrib of the plant comprising the stem or midrib, and often referred to as tobacco petioles, are the preferred tobacco materials. Tobacco stalks contain lesser amounts of pectinaceous materials but can also be employed.

In the first treating step of the process of our invention, tobacco pectins are liberated from pectinaceous materials in tobacco by reacting the pectinaceous materials with a reagent which, under the conditions of the reaction, is reactive with the calcium (and/or magnesium) contained in them to form a compound or product having a lower calcium ion, and in the case of magnesium, magnesium ion, concentration[1] in the treating solution than the naturally occurring calcium (or magnesium) pectate. This reagent may, for convenience, be hereinafter referred to as a "cross-link destroying reagent."

The reaction is generally represented by Equation I, which illustrates the reaction of one type of tobacco protopectin (a calcium salt of a polymer of galacturonic acid), wherein calcium cross-links are present with the reagent of the present invention. In the equation, R may be hydrogen, in which case the product is pectic acid, or R may be a monovalent inorganic cation, such as sodium, potassium or ammonium, in which case the product is a soluble pectate.

[1] By concentration, we mean concentration or activity as set forth in Glasstone, "Textbook of Physical Chemistry," 2nd Edition, p. 554, D. V. Nostrand Company, Inc.



In one embodiment of this invention, the reagent, which can be, and preferably is, in aqueous solution, acts by forming a precipitate with the calcium or magnesium, in which case, it can be a water-soluble monovalent metal salt of the formula M_xX wherein M is a monovalent inorganic cation, x is an integer having a value of 1, 2, or 3 and X is an anion which may be monovalent or polyvalent, such that the calcium salt of the formula Ca_yX_z is essentially insoluble in the treating solution and y and z are integers corresponding to the functionality of X. Monovalent cations which are effective include the alkali metals such as sodium, potassium and lithium, and also include such monovalent cations as ammonium, and substituted ammonium ions $(NR_4)^+$, where R=aryl or alkyl. The anion portions of the molecule may be $CO_3^{=}$, $PO_4^{=}$, $HPO_4^{=}$, $H_2PO_4^{-}$, and the like. For example, the compound MnX could be sodium carbonate, Na_2CO_3, since sodium is a monovalent inorganic cation and calcium carbonate is essentially water insoluble. Additional representative examples of precipitating agents are the orthophosphates, metaphosphates and carbonates of sodium, potassium, lithium and ammonium. In the case of the orthophosphates, the anion portion of the molecule may be either $PO_4^{=}$, $HPO_4^{=}$, or $H_2PO_4^{-}$. Specifically, for example, when ammonium orthophosphate is used, the precipitate is calcium and/or magnesium ammonium phosphate. The pH of this reaction should be between about 5.8 and 10 and the temperature may be as high as 400° C. but should, preferably, be between about 25° C. and about 135° C. for a period of from about 1 minute to about 24 hours. Preferred precipitating agents which may be employed are the alkali metal carbonates, for example, sodium carbonate and potassium carbonate. Particularly preferred precipitating agents which may be employed are the alkali metal phosphates and, most particularly, the alkali metal orthophosphates and the ammonium orthophosphates, such as the ammonium phosphates and ammonium orthophosphate, sodium orthophosphate, potassium orthophosphate, sodium dihydrogen orthophosphate, ammonium dihydrogen orthophosphate, potassium dihydrogen orthophosphate, diammonium monohydrogen orthophosphate, disodium monohydrogen orthophosphate and dipotassium monohydrogen orthophosphate.

In a second embodiment, the cross-link destroying reagent acts by sequestering the calcium or magnesium, thereby removing the calcium or magnesium atoms by forming a complex therewith. Suitable reagents of this type include any sequestering agent which will form a complex or chelate with the calcium and/or magnesium, thereby removing the calcium and/or magnesium and making them unavailable for cross-linking with the pectin. Illustrative of such sequestering agents are ethylene-diamine-tetraacetic acid and similar amino acids, alkali

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metal polymetaphosphates such as tetrametaphosphates, hexametaphosphates and trimetaphosphates, pyrophosphates and tripolyphosphates, such as sodium hexametaphosphate, tetrasodium pyrophosphate and pentasodium tripolyphosphate. The mechanism which occurs when a sequestering agent is employed is the formation of a chelate; calcium and magnesium ions are no longer available to combine with the pectate ions in solution. Many naturally occurring amines and peptides are also effective as sequestering agents for calcium and/or magnesium. Representative examples include alanine, aspartic acid, glycine, glycyl glycine, glutamic acid, serine, tyrosine and diiodo-l-tyrosine. Amino acids that are effective as chelating solubilizing agents include beta alanine, N,N-diacetic acid; amino barbituric acid, N,N-diacetic acid; 2-amino-benzoic acid, N,N-diacetic acid; beta-aminoethylphosphonic acid, N,N-diacetic acid; beta-aminoethylsulfinic acid, N,N-diacetic acid and ethylenediamine-tetraacetic acid. The pH of this reaction should, preferably be between about 8 and about 10 and the temperature should, preferably be between about 0° C. and about 145° C. for a period of from about 1 minute to about 24 hours.

A cross-link destroying reagent may also function partially as a sequestering reagent, in accordance with the first embodiment of this invention, and partially as a sequestering reagent in accordance with the second embodiment. Such a reagent, for example, is diammonium monohydrogen orthophosphate (DAP), which is a particularly preferred reagent in accordance with the invention.

In a third embodiment, the cross-link destroying reagent is an acid wash which forms the released but insoluble free pectic acid and soluble calcium and magnesium salts. Generally the acid wash will comprise an inorganic acid, such as hydrochloric acid, phosphoric acid, sulfuric acid or a similar acid, which will form soluble calcium and magnesium salts under the following conditions. Hydrochloric acid and sulfuric acid are particularly preferred. The acid may be employed as 0.25 N to 5.0 N solutions. It is preferably employed as 0.5 N to 1.0 N solutions. The exact dilution and amount to be employed will vary with the particular acid which is used, it only being necessary that sufficient acid be present to convert the calcium and magnesium present in the tobacco being treated to the calcium and magnesium salts of the acid. The acid treatment is preferably conducted at a temperature of from about −1° C. to about 50° C. The acid treatment comprises reacting the tobacco parts with the acid until the resulting mixture has a pH of from about 1.0 to about 6.5. Preferably, the pH is brought to from about 1.0 to about 3.7, the most desirable pH being between 1 and 2.5. This treatment will generally be conducted from about 10 minutes to 24 hours, depending in part on the size of the tobacco particles. The acid conditions which are necessary for this embodiment of the invention may be achieved by the use of ion exchange resins which may be used, with suitable recycles, to obtain the desired pH of the solution during treatment. The ion exchange resins may be used and regenerated in accordance with conventional practices for such resins.

Preferably, the mixture resulting from the acid treatment is then washed with water. This water wash step is preferably conducted at a temperature of from about 15° C. to about 35° C. and, preferably, distilled water is employed. When this wash step is employed, sufficient water should be used to remove the calcium and magnesium salts of the acids, which salts are formed in the above-described treatment, thus, there should be at least 2 volumes of water per volume of the mixture resulting from the acid treatment. The wash water is separated from the tobacco by any suitable means, for example, by conducting the wash in a centrifuge, filter press, Buchner funnel, or any other apparatus from which liquids can be substantially removed from solid materials.

In summary, in the first step of the first embodiment of this invention the treating agent Z–R attacks the calcium and/or magnesium cross-links of tobacco protopectin and forms a precipitate which is a salt of calcium and/or magnesium, thus removing the calcium and/or magnesium from the protopectin and from the solution. In the first step of the second embodiment, the treating agent Z–R is a sequestering agent which forms a chelate of the magnesium and/or calcium from the tobacco protopectin and makes the calcium and/or magnesium unavailable for recombining with the pectins. With certain reagents, such as DAP, the first step of the present process may comprise a combination of the mechanisms of the first embodiment of this invention and the mechanism of the second embodiment of this invention. In the first step of the third embodiment of this invention, the treating agent Z–R is an acid which attacks the calcium and/or magnesium cross-links of the tobacco protopectin and forms the soluble calcium and/or magnesium salts, which are then washed away from contact with the pectins.

In the first step of each of the first two embodiments of the invention, the pectin which results is in condition for release from the tobacco cell structure, R in Equation 1 being a monovalent inorganic cation such as sodium. In the first step of the third embodiment, the insoluble pectic acid resulting from the acid treatment must be reacted with an alkaline material before it is in condition for release from the tobacco cell structure.

The acid treated pectins are placed in condition for release by bringing the mixture resulting from the acid treatment, and preferably, after the water wash described above, to a pH of from about 5.0 to about 10.5 and, preferably, from about 6.3 to about 8.5, by the addition of an alkaline material. Suitable alkaline materials include ammonium hydroxide and alkali metal hydroxides, for example, sodium hydroxide, potassium hydroxide and lithium hydroxide, and alkali metal salts, such as sodium bicarbonate, sodium carbonate, sodium phorphate, and similar salts to convert the pectic acid to a soluble form. The alkaline material may be any water-soluble compound containing a monovalent inorganic cation and capable of producing hydroxide ions when dissolved in water. The temperature of this step may be from about −1° C. to about 45° C., but is, preferably, from about 15 to about 35° C. The alkaline material is preferably in the form of solid particles or in the form of a solution having a concentration of from about 5 to about 50%.

Once the tobacco pectins have been liberated from the tobacco, by the removal of the calcium and magnesium cross-links, they should be released from the interstices of the tobacco. That is, they will be made available to the solution or suspension or, in certain instances, they will be merely deposited on the surface of the tobacco particles. This comprises the second step of the process of the present invention. In embodiments 1 and 2 of the invention, this release or second step may be accomplished concurrently with the first step by reacting with the solution of the treating reagent. In embodiment 3, however, as indicated above, the insoluble pectic acid resulting from the treatment should be reacted with an alkaline material before it can be released. In such event, the release may be concurrent with the addition of the alkaline material due to a washing action. In any case, additional treating liquid or water may be used to effect the release through a washing action of the treated tobacco particles.

In accordance with the third step of our process, the liberated and separated tobacco pectins can next be precipitated or deposited in a relatively free form (as compared with the tobacco pectins as they were originally present in the tobacco), from the treating solution, for example, by being formed into the insoluble pectic acid or into an insoluble salt of pectic acid or by the action of a water-miscible solvent, such as acetone or ethyl alcohol to cause a water-soluble salt of pectic acid to go out of so-

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solids. In the case of tobacco pectin solutions such as solutions of sodium and potassium pectinates and/or pectates, this can be accomplished by acidifying the solution until the pectins precipitate or by adding a gelation agent, such as an alcoholic solution, preferably having a pH of from about 1 to 9 and, preferably, from about 1 to 5. The pH of the alcoholic solution can be regulated by the addition of a mineral acid such as HCl, to the alcohol. Although the preferred gelation agent is ethanol, any water-miscible organic solvent having up to about 10 carbon atoms may be employed, for example, a ketone, such as acetone, or a diether, such as dioxane. Water-immiscible solvents such as ether, for example, ethyl ether, can be used, if combined with a water-miscible solvent, such as acetone.

The tobacco pectins can be recovered by concentrating the solution or suspension in which they are present until they precipitate. This precipitate might also be characterized as an intractable mass, since the pectin solution, upon concentration, generally becomes progressively more viscous until it finally ceases to leave a deposit in a glassy solid state.

While the tobacco pectins can be separated and purified before use as a binder in reconstituted tobacco sheets, they are preferably employed just as they are produced in situ, i.e., in combination with the treated tobacco plant parts from which they were obtained, the entire combination comprising a binder for reconstituted tobacco sheets or, under some circumstances, the entire combination comprising essentially the entire components of a reconstituted tobacco sheet. By using the entire mixture, no original tobacco flavors are lost, all of the tobacco is employed, and no expensive and time-consuming refining operations are necessary.

Although it is not necessary, the thixotropic properties of solutions containing soluble pectins can be adjusted in the preparation of cured sheet by the addition of such materials as calcium chloride. If any complex or precipitate formed in the first step of this process is present with the insoluble pectates, the thixotropic properties of the mixture can also be adjusted by adjusting the pH to precipitate calcium and magnesium pectates.

A preferred preliminary step, in accordance with the present invention, comprises washing the tobacco plant parts which are preferably ground or cut to a relatively small size, with cold water. This water wash serves to remove impurities which might otherwise hinder the subsequent treatments in accordance with the present invention. It is particularly desirable to employ such a cold water washing step when alkali metal carbonates are employed as the gelling agent in the first step. Generally, sufficient water should be employed during such a water wash operation to cover all of the tobacco plant parts present. The temperature of the water may be between −1° C. and 100° C., but is preferably about 20° C., and the water wash should generally continue for a period of from about ½ to 2½ hours. Agitation during the wash is desirable, but not necessary. After the water wash has been completed, the water can be removed from the tobacco parts by filtration, centrifugation or other suitable means.

As discussed supra, a particularly preferred embodiment of the present invention involves the use of an ammonium or substituted ammonium orthophosphate, such as diammonium monohydrogen orthophosphate (DAP), for the release of the tobacco pectins. The DAP will, generally, be added to the tobacco plant parts, which may, for example, be bright tobacco parts, burley tobacco parts, or a mixture of the same, in an aqueous solution. The concentration of the DAP in the aqueous solution is not critical, but will, generally, be in the range of 0.5–3.0% by weight. The DAP and water may be added separately to the tobacco. The amount of DAP should, preferably, comprise from about 0.01 to about 0.5 part, and, most preferably, from about 0.05 to 0.33 part (by weight) per part of tobacco being contacted. A humectant, such as glycerin or triethylene glycol, may be present, if desired, at about 0.5 to 1 per part of DAP. The temperature during the DAP treatment of the tobacco may vary between room temperature and about 190° F. or higher, depending on the type of tobacco being treated. Under pressure, even higher temperatures may be employed. The pH of this mixture is, preferably, maintained at a value about 7.1 to 9.0, which may conveniently be accomplished by the addition to the solution of concentrated aqueous ammonia. By agitating or stirring such a mixture under the above-described conditions for from about 1 minute to about 1 day, and, preferably, from about 1 hour to about 5 hours, the tobacco pectins are liberated, released and deposited in the tobacco plant parts to form a binder composition. The resulting mixture may then be refined, for example, in a disk refiner, until substantially all the pulp (in excess of the water present), can be shaken through a screen of approximately 18 mesh to produce a binder composition which is ready for use in the manufacture of reconstituted tobacco sheets.

The term "tobacco pectins" as used throughout this specification means "liberated tobacco pectins" and comprehends pectins which have been freed or liberated from tobacco and are, therefore, not bound into the tobacco structure, as differentiated from the insoluble, naturally-occurring protopectins which are found into a plant cell structure. The term includes the free pectinic or pectic acid, as well as soluble salts such as the sodium, potassium, ammonium, pectates and pectinates, and insoluble salts such as the calcium and magnesium pectates and pectinates depending on what method is employed to liberate and obtain them from the naturally occurring insoluble protopectins.

The tobacco pectins produced or liberated in situ or isolated by means of this invention can be used as the sole binder material for reconstituted tobacco, i.e., no other materials need be added to make the sheet. They can be sprayed, extruded or cast, thus facilitating application onto a moving belt carrying tobacco dust. Under proper conditions of formulation and processing, reconstituted tobacco made with the tobacco pectins produced by this invention exhibit excellent physical and aromatic properties. The ultimate tensile and wet strengths of the reconstituted tobacco are good. While no other materials need be added to the pectinaceous binder, other materials can be added, if desired. For example, organic acids and preservatives which may in themselves be of tobacco origin, may be added. Plasticizers, such as glycols and polyglycols, and humectants, such as glycerin, may also be added, if desired. In addition, the gel strength of the tobacco pectins can be regulated by partial precipitation to control such rheological properties as viscosity, fluidity and elasticity. Other additives or dispersants may be added in small amounts to regulate slurrying qualities, provided, however, that such substances are not added in large enough quantities to adversely affect the flavor or aroma of the final product. Furthermore, the tobacco pectins can be combined with water-soluble gums or water-dispersible gums commonly used as binders for tobacco sheets such as methyl cellulose, sodium carboxymethyl cellulose, guar gum, locust bean gum, or alginates, although it is preferred to minimize or eliminate such additions in order to obtain a product which most closely resembles natural tobacco.

The product from treating the tobacco plant parts in accordance with the methods of the present invention may be cast directly and dried and cut into particulate material similar in physical form to ordinary smoking tobacco and so used, preferably mixed with tobacco leaf cut or shredded in the usual manner. The product may be cast in sheet form, in blocks or as threads or other shapes, as desired. An important use, however, of the prepared composite slurry or easily molded isolated pectinaceous mass is as a binder for ground tobacco and for the making of corresponding tobacco products suitable

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for smoking. Sheet material of widely different properties may be formed by suitable variations in the manner of forming. One method and product comprises flowing the composite slurry onto a moving belt and applying a layer of dry, ground or fragmented tobacco to the wet adhesive surface. If desired, there may be first applied to the belt a layer of the tobacco, followed by a layer of the binder, and then a top layer of the tobacco. Various additives may be included with the ground tobacco such as flavorants, plasticizers and aromatic substances. The web is ultimately dried and then suitably moistened and rolled up. Such methods of forming continuous sheets are known generally in the art and the details need not be further described. Representative of this procedure is the apparatus and method disclosed in U.S. Patent 2,734,513.

Another method of forming a reconstituted tobacco product, with the slurry of the isolated tobacco pectins as a binder, comprises mixing ground tobacco thoroughly therewith into a mass of dough-like consistency and then casting the mass in sheet form onto a moving belt surface followed by drying and remoistening in accordance with the known procedures. Representative of this procedure is the apparatus and method disclosed in U.S. Patents 2,703,171 and 2,794,734. Obviously, the reconstituted tobacco may also be formed by molding or other suitable means.

A particularly preferred aspect of the present invention comprises employing as a binder or directly, the mixture of tobacco and tobacco pectins which have been produced in situ, without any separation steps and without the necessity for any additional adhesive materials.

The following examples are illustrative:

Example 1

Tobacco stems (10 parts) were covered with cold water and leached for ½ hour. The water was then decanted and discarded. A cooking solution, made from 1 part of sodium carbonate dissolved in 60 parts of water, was added to the leached parts. This mixture was heated at the boiling point for 30 minutes at atmosphere pressure and then for 20 minutes at 20 p.s.i.g. In the course of this treatment the tobacco pectins were dissolved from the tobacco parts. The entire wet mass (pulp mixture) was dried and ground in a Waring Blendor so that it would pass through a 30 mesh screen. The resultant material had gel-like properties and was thixotropic in nature.

To this material was added 2 parts of glycerin, to serve as a humectant. The pH of the resulting mixture was adjusted to 7 by the addition of a solution of 10% hydrochloric acid. One gram of sodium carboxymethyl cellulose (CMC) was added to the mixture, giving a proportion of about 1 part of CMC to 10 parts of total solids in the mixture. As it was desired to use this mixture as a sprayed-on type binder, the CMC was employed in order to adjust the spraying qualities. The mixture of (a) treated tobacco plant parts, including the liberated tobacco pectins, (b) the sodium carboxymethyl cellulose and (c) the glycerin had a viscosity which was suitable for spray application of the mixture as a binding material for reconstituted tobacco. This material was sprayed on top of an undercoating of tobacco dust, of an 80 mesh size, which had been dusted on a wet belt. Another coat of the tobacco dust was laid on top of the binder. In general an apparatus similar to that described in U.S. Patent 2,734,513 was employed. As in said patent, the reconstituted sheet was dried and was then humidified to the desired moisture content. The control in Table I was made in a conventional manner, using a relatively large amount of CMC in proportion to the pulped washed tobacco stems. The test data as observed are recorded in Table I.

TABLE I

Sample	Sheet 1	Sheet 2	Sheet 3	Control
Part CMC [1]	1:10	1:10	1:20	1:5
Moisture (percent) [2]	12.3	12.1	12.7	12.4
Tensile, g./in. [3]	8.0	7.9	4.8	9.2
Elastic Tensile, g./in. [4]	7.1	7.0	4.1	7.6
Elongation (percent) [5]	2.3	2.4	3.6	1.5
Bursting Point, lbs. [6]	1.6	1.6	1.9	1.7

[1] Part CMC per parts of total tobacco solids in the slurry (including tobacco pectins).
[2] The percentage of moisture contained in the entire sheet (wet basis).
[3] The breaking strength of a 10 cm. test strip which is 1 inch wide, as average of 10 strips.
[4] One-half that force required by application of a 500 gram weight for 30 seconds.
[5] The percent elongation at breakage on the Instron test machine.
[6] Free diameter ball bursting value of a single test strip, in air.

The reconstituted tobacco sheets made pursuant to this example were shredded and made into cigarettes. The test cigarettes, as well as control cigarettes made from standard reconstituted tobacco sheet, were submitted to a smoking panel for subjective testing. The panel found that the smoke of the cigarettes made by the method of this invention was significantly less harsh than the smoke obtained from the control cigarettes. A pleasing vanillin odor was found to be transmitted into the aerosol phase of the smoke from the test cigarettes made employing reconstituted tobacco made by the present invention.

Example 2

Fifty grams by weight of burley tobacco stems were thoroughly washed in about 5 liters of cold water for three hours. The washed tobacco stems were then mixed with 500 grams of water having dissolved therein 5 grams of sodium carbonate. The resultant mixture, containing about 8% solids, was steamed under atmospheric pressure for 30 minutes and under a pressure of 20 p.s.i.g. for an additional 20 minutes. At the end of this time, the mixture was allowed to cool and the liquid was separated from the solid materials. The solid materials were treated in a cider press to recover as much as possible of the remaining liquid. The liquid was added to a previously prepared coagulant bath which consisted of ethanol and hydrochloric acid, in an amount to adjust the pH of the coagulant to about 1.0. The resulting mixture (which had a pH of about 3.0) was alternately stirred and allowed to settle for a period of two hours. At the end of this time the mixture was strained through a cloth sieve and the filtrate was discarded leaving a solid, gel-like mass, consisting essentially of pectinaceous materials combined with approximately 10 parts of liquor. The yield of pectinaceous materials was 15%, based upon the dry weight of the starting tobacco plant parts. The mass was observed to be thixotropic, soluble in water at a pH of about 6 and soluble in a sodium carbonate solution.

The tobacco pectins thus isolated were quite impure and had a color characteristic of tobacco. The solid mass was then redissolved in a sodium carbonate solution and the resultant solution was poured into an acidified ethanol bath similar to the coagulant bath used earlier. The recoagulated solid was obtained by filtration and dried in an oven at 105° C. The dried tobacco pectins were in the form of an opaque, substantially colorless sheet. When the sheet was pulverized, a white powder was obtained having a distinct and pleasing odor similar to vanillin.

Ten grams of the dried tobacco pectins prepared above were swelled in 100 ml. of cold water. The mixture was then added to a slurry consisting of 8.5 grams of refined kraft pulp dispersed in 200 ml. of water. The resultant mixture was homogenized in a Waring blendor. Four grams of glycerin were then added to the mixture to serve as a humectant. The resulting mixture was cast into a binder film, using a Gardiner casting knife set to produce a sheet having a wet thickness of 50 mils. The physical properties of this sheet, after drying, were tested and were found comparable to the properties of conventional binder films. The physical test data are given in Table II. The control material was made by mixing pulp, which had

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been refined with NaOH, with sodium carboxymethyl cellulose (CMC) and a humectant, as is shown in Table II.

TABLE II

	Test binder-film	Control binder-film
Binder (dried)	(1)	(2)
Basis weight, gms./ft.²	6.3	5.84
Moisture, percent	14.3	14.0
Tensile modulus	0.48	0.60
Tensile strength, p.s.i.	4.3	4.5
Elongation, percent	6.1	6.1
Work coefficient, gram cm. per sq. cm.	37.97	31.8

(1) Dried tobacco pectins extracted from tobacco petioles.
(2) CMC (sodium carboxymethyl cellulose) and pulp, as prepared by Example 1 and containing about 40% CMC.
* Work coefficient is proportional to the product of the tensile strength and the elongation.

Example 1

One hundred grams of burley tobacco stems were covered with distilled water, leached for 30 minutes and then drained. The washed stems were then mixed with a solution containing 9.3 grams of diammonium monohydrogen orthophosphate in 600 ml. of water. The resultant slurry was heated for 1 hour at a temperature of from 90 to 100° C. The pH of the reaction mixture, after completion of the reaction, was about 7. The entire reaction mixture was homogenized in a Waring blender. The solids content was determined to be 5% by weight.

Two grams of glycerine, to serve as a humectant, were blended with 200 grams of the above reaction mixture. A film of 50 mils wet-thickness was cast from this mixture. The extruded mixture was found to have the property of being able to slide on a surface even when newly cast. After the film was completely dried, it was found that it could be peeled off a plate by hand, while still partially wet and then hung up to dry like wet cloth. This property provides definite advantages in the manufacture of tobacco products.

Physical tests were carried out on the dried film thus prepared and were carried out on a control film made by using conventionally pulped tobacco plant parts with sodium carboxymethyl cellulose (CMC) as the binder. A film as binder as made in this example was burned and was found to give a pleasant aromatic smoke. Testing data are given in Table III.

TABLE III

	Test binder-film	Control binder-film
Binder	(1)	(2)
Basis weight, gms./ft.²	6.9	5.8
Moisture, percent	14.3	14.0
Tensile modulus	0.52	0.57
Tensile strength, p.s.i.	4.3	4.3
Elongation, percent	13.1	6.1
Work coefficient, gram cm. per sq. cm.	51.5	31.6

(1) Dried tobacco pectins extracted from tobacco petioles and formed as a film.
(2) CMC plus pulp.
(3) Kg. per gm. of basis wt. (ft.).
(4) Kg. in. per g. in. (ft.).
* Work coefficient is proportional to the product of the tensile strength and the elongation.

Examples 4–10

In each of these examples, burley tobacco stems were covered with distilled water, leached for 30 minutes, and drained. One hundred grams of stems were treated with the agent indicated in Table IV. The agent was dissolved in 600 ml. of distilled water in an amount sufficient to make up a 10% (by weight) solution. The leached tobacco stems were placed in said solution and heated in a steam bath for 1 hour at a temperature of from 90 to 100° C. (Except for the example in which Versene was employed. That example was conducted at room temperature.) A five ml. aliquot was taken from the resulting mixture and mixed with 20 mls. of ethyl alcohol in a graduated cylinder whereby a precipitate of tobacco pectins was obtained. The amount of precipitate in the graduated cylinder was observed. Each of these precipitates of tobacco pectins was suitable for use in the preparation of a reconstituted tobacco sheet in accordance with the methods taught by the present invention. The results obtained in these experiments are presented in Table IV below.

TABLE IV

Reagent	Volume of coagulated tobacco pectins
Pentasodium tripolyphosphate	15
Sodium hexametaphosphate	5
Diammonium monohydrogen orthophosphate (DAP)	15
Trisodium orthophosphate	15
Disodium monohydrogen orthophosphate	15
Dihydrogen monosodium orthophosphate	5
Versene (tetrasodium salt of ethylenediaminetetraacetic acid)	15

Example 11

One hundred grams of burley tobacco stems were covered with distilled water, leached for 30 minutes and drained. A solution of 10 grams of Versene (tetrasodium salt of ethylenediaminetetraacetic acid) in 600 ml. of water was added to the stems and the mixture was made basic with aqueous sodium hydroxide. The resulting mixture was then heated for 1 hour at room temperature. The entire mixture (a pulp) was homogenized in a Waring Blendor and the solids content was determined to be 5% by weight. A 200 gram portion of the pulp, thus prepared, was combined with 2 grams of glycerine, as a humectant, and then cast into a film of 50 mils thickness by the use of a Gardiner casting knife. The resultant sheet was found to have satisfactory physical properties and, upon burning, exhibited a very pleasing aroma.

Example 12

Green bright tobacco leaves were soaked in isopropanol to remove sugars and chlorophyll. The midrib was stripped out of the leaves leaving the web. One thousand grams of the web (82 grams on a solids basis) were placed in a porcelain bucket and covered with boiling distilled water containing 9.3 grams of diammonium monohydrogen orthophosphate (DAP). The DAP was added on the basis of 10 grams of DAP per 100 grams of stems (which contained 12% moisture, by weight). The resulting mixture was boiled at low heat for one hour. The juice was then first expressed from the mixture by hand. Solids, for example cellulose and sand, were then further separated from the juice by centrifuging. The clear juice from both operations was then mixed with 70% ethanol to form a gel, which was then squeezed out in cheese cloth. The gel was transferred to a Buchner funnel where it was washed first with acetone and then with ethyl ether. Finally, the gel was placed in a vacuum desiccator and dried. The gel was suitable for use in the preparation of a reconstituted tobacco in accordance with the teachings of the present invention.

Example 13

Burley tobacco stalks were separated into cortical tissue, woody tissue, and pith. A sample of each was steam cooked with 10% sodium carbonate for 30 minutes at atmospheric pressure and for 20 minutes at 20 p.s.i.g. After this cooking, the woody tissue was still hard and could not be pulped in a Waring Blendor. The cortical tissue and pith were soft and pulpable. Each of these latter two preparations were suitable for use in the preparation of reconstituted tobacco.

Example 14

Coarse ground bright stem tobacco fines (30 grams) were washed thoroughly in cold water, and then placed in a boiling aqueous solution of 3 grams of diammonium monohydrogen orthophosphate and cooked for 5 minutes. The mixture was then placed in a Waring Blendor.

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After a very short period in the blender, the mixture was converted to a viscous, fine, impalpable slurry, wherein the tobacco stem particles had been completely separated to units of cellular size. This impalpable mass was suitable for use as a binder in reconstituted tobacco. When remixed with some of the cold water washings removed in the first step, the impalpable mass immediately became viscous and eventually jelled to a soft mass.

For further comparison, a similar sample of coarse ground bright stem fines (30 grams), was dispersed in boiling water and cooked for about 30 minutes with three grams of diammonium monohydrogen orthophosphate. The pH of this mixture was brought to a value of 7.1 by the addition of the mixture of 30% aqueous ammonia. The granules of tobacco could then be refined to a palpable pulp, similar to the soft mass produced in the experiment described in the first part of this example.

Example 15

Burley tobacco stems (½ pound) were covered with distilled water, allowed to stand several hours and the water decanted. This step was repeated several times and finally the stems were covered with dimilled water containing 10 ml. of concentrated HCl and left overnight at 24° C.

After standing overnight the acidic water was decanted and the stems washed repeatedly free of HCl until the wash water gave no precipitate when treated with silver nitrate solution. The stems were then covered with distilled water containing 15 grams of sodium carbonate and left overnight at 24° C. The pH of the sample, the following morning was 8.8. The stems were swollen and soft and were easily disintegrated with the fingers and the mixture could be homogenized and employed, as a binder in reconstituted tobacco.

Example 16

The apparatus employed in this experiment was large scale equipment consisting a 200 gallon conical bottom, open top, stainless steel tank, fitted with a Cowles high speed mixer.

One hundred and forty-eight gallons of water were placed in the tank and heated to a temperature of 207° F. One hundred and forty-eight pounds of bright tobacco (milled to pass a 6 mesh per inch sieve) stems were added, while operating the Cowles mixer at a low speed. Almost immediately after the addition of the tobacco, nine and one-quarter pounds of diammonium monohydrogen orthophosphate (technical grade) were added to the mixture. Ammonia (assaying 28%, by weight NH_3) was added to adjust the pH to 7.1. The mixing speed was increased to 3700 r.p.m. After a period of 3 minutes the temperature of the mixture was 194° F. Most of the particles in the mixture were soft enough to be smeared by hand and the mixture had a jelly-like consistency. The mixing was continued for 1 hour to obtain the highest possible state of disintegration, although a 15 minute period appeared to be sufficient for this purpose.

The viscosity of the mixture was found to be 10,400 cps. and its solids content was found to be 8.05% by weight.

This mixture was employed as a binder, being pumped through a filter to storage tanks and subsequently sprayed on tobacco by the method and equipment described in U.S. Patent 2,734,515. It was applied at the rate of 3 grams per square foot to form a reconstituted tobacco sheet having the following physical properties:

Basis weight, gms./ft.² (1)	10.3
Moisture content (percent by weight)	13.0
Tensile, kg./in.	0.82
Fold tensile, kg./in.	0.82
Work-to-break, gm. cm./ft.² (2)	16.0

(1) Weight of all the ingredients, including tobacco pulp, essentially free of moisture.
(2) Computed by an integrator attached to the Instron Tensile Tester.

Example 17

Burley tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then dried and ground and used to make a binder as follows:

One hundred parts of water were brought to a temperature of 195° C., and to this were added:

7.00 parts by weight (dry basis) of the washed burley stems,
1.05 parts diammonium phosphate, and
0.70 part of glycerin, as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 7.1 but no higher than 9.0.

The mixture was then stirred for one hour and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 18

Burley tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then used directly in the wet condition to make a binder as follows:

One hundred parts of water were brought to a temperature of 195° C., and to this were added:

7.00 parts by weight (dry basis) of the washed burley stems,
1.05 parts diammonium phosphate, and
0.70 part of a glycerin, as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 7.1 but no higher than 9.0.

The mixture was then stirred for one hour and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 19

Burley tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then dried and ground and used to make a binder as follows:

One hundred parts of water were brought to a temperature of 195° C., and to this were added:

7.00 parts by weight (dry basis) of the washed burley stems,
1.05 parts diammonium phosphate, and
0.70 part of triethylene glycol (TEG), as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 7.1 but no higher than 9.0.

The mixture was then stirred for one hour and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

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Example 20

Burley tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then used directly in the wet condition to make a binder as follows:

One hundred parts of water were brought to a temperature of 195° C., and to this were added:

7.00 parts by weight (dry basis) of the washed burley stems,
1.05 parts diammonium phosphate, and
0.70 part of triethylene glycol (TEG), as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 7.1 but no higher than 9.0.

The mixture was then stirred for one hour and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 21

Bright tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then dried and ground and used to make a binder as follows:

One hundred parts of water were brought to a temperature of 100° C., and to this were added:

7.00 parts by weight (dry basis) of the washed bright (flue cured) stems,
1.05 parts diammonium phosphate, and
0.70 part of glycerin, as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 8.0 but no higher than 8.5.

The mixture was then stirred for four hours and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 22

Bright tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then used directly in the wet condition to make a binder as follows:

One hundred parts of water were brought to a temperature of 100° C., and to this were added:

7.00 parts by weight (dry basis) of the washed bright (flue cured) stems,
1.05 parts diammonium phosphate, and
0.70 part of glycerin, as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 8.0 but no higher than 8.5.

The mixture was then stirred for four hours and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 23

Bright tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then dried and ground and used to make a binder as follows:

One hundred parts of water were brought to a temperature of 100° C., and to this were added.

7.00 parts by weight (dry basis) of the washed bright (flue cured) stems,
1.05 parts diammonium phosphate, and
0.70 part of triethylene glycol (TEG), as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 8.0 but no higher than 8.5.

The mixture was then stirred for four hours and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 24

Bright tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then used directly in the wet condition to make a binder as follows:

One hundred parts of water were brought to a temperature of 100° C., and to this were added:

7.00 parts by weight (dry basis) of the washed bright (flue cured) stems,
1.05 parts diammonium phosphate, and
0.70 part of triethylene glycol (TEG), as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 8.0 but no higher than 8.5.

The mixture was then stirred for four hours and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 25

In the acid wash treatment of tobacco parts for removal of alkaline earth minerals of the tobacco pectin, it is necessary to use quite large volumes of water if it is desired to reduce the soluble anion content of the product to a very low level. In this case, the use of a cation exchange resin in a closed loop system with the tobacco parts can make it possible to conduct the process with limited amounts of water and acid.

The use of a resin (such as Dowex 50 W) in this way does not at all alter the principles of this form of treatment since the resin merely serves as a convenient reservoir of acidity, continuously reconditioning effluent from the stems for reuse in the extraction.

In the following example, the use of a cation exchange resin permits the treatment of stems with a small fraction of their natural nitrate ion content. Hydrochloric acid is used at intervals to regenerate the resin, but never directly contacts the tobacco.

The apparatus used for ion-exchange extraction of calcium from tobacco stems consisted of a 10 inch diameter washing column fitted with a 20 mesh screen to support the charge of stems and a 6 inch diameter Pyrex column filled with 13 pounds of Dowex 50 W-8 acid-form ion exchange resin beads. The valves and piping permitted a pump to be used either for recycling waters from the stem charge through the column or for rep-

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ately regenerating the resin with hydrochloric acid solution.

In operation, fifty pounds of stems were placed in the washing column and rinsed with cold water until they were nearly free of water-soluble material. Then, the effluent waters were diverted through the ion exchange bed, returning by gravity to the top of stem charge. When the aqueous extract from one pound of burley stems was added to the recycling waters, a pH of between 1.5 and 2.0 was soon attained. During the treatment, the waters flowing from the resin column were monitored for their calcium content. (An appreciable precipitate from 50 ml. of resin effluent treated with ammonium oxalate and made basic with ammonia indicated that the resin was saturated with calcium and needed to be regenerated with hydrochloric acid.) After sixteen hours of operation, and one regeneration of the ion exchange resin, the treatment was considered complete. A subsequent analysis showed that the calcium content of the stem had been reduced to less than one-sixth of the starting value of 4.5% CaO. The stems were rinsed twice with cold distilled water, pressed lightly, and redried in a tobacco processing oven to a final moisture content of about 4%, and ground to pass a 40 mesh screen. When a small sample of this powder was stirred with a little cold ammonia, it dispersed immediately, forming a stiff paste.

The resulting material was then employed as a binder for tobacco powder to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 26

Twenty grams of tobacco fines were washed in 1 liter of distilled water to get rid of the tobacco solubles. The fines were then admixed with 1 N hydrochloric acid until the mixture had a pH of 1.35. The pH was checked on a Beckman pH machine and the curves were also plotted against a titration of distilled water.

After titration, the acid was washed off the fines with three 400 ml. portions of distilled water. The fines were redispersed in 400 ml. of distilled water and sodium bicarbonate was added to bring the pH to 7. The slurry was cast at a thickness of 30–50 mils and dried to form a sheet and the resulting sheet was used as the adhesive binder in sandwich-type reconstituted tobacco sheet.

Example 27

Twenty grams of tobacco fines were washed in 1 liter of distilled water to get rid of the tobacco solubles. The fines were then admixed with 1 N hydrochloric acid until the mixture had a pH of 1.35. The pH was checked on a Beckman pH machine, and the curves were also plotted against a titration of distilled water.

After titration, the acid was washed off the fines with a 400 ml. portion of distilled water. The fines were redispersed in 400 ml. of distilled water and sodium bicarbonate was added to bring the pH to 7. The slurry thickened to a viscous mass which is evidence that the pectin had been solubilized. The slurry was cast at a thickness of 30–50 mils and dried to form a sheet and the resulting sheet was used as the adhesive binder in a sandwich-type reconstituted tobacco sheet.

Examples 28–30

In each of these three examples, 20 gms. of tobacco fines were washed in 1 liter of distilled water to get rid of the tobacco solubles. The fines were then admixed with 1 N hydrochloric acid until the mixture had a pH of 1.35. The pH was checked on a Beckman pH machine and the curves were also plotted against the titration of distilled water.

After titration, the acid was washed off the fines with three 100 ml. portions of distilled water. The fines were redispersed in 400 ml. of distilled water and a neutralization agent, as indicated in Table V, was added to bring the pH to the value shown in Table V. The slurry in each case thickened to a viscous mass which is evidence that the pectin had been solubilized. The slurry was cast at a thickness of 30–50 mils and dried to form sheets. The binders formed in this way were tested to determine their physical properties. These properties are shown in Table V below. The binder was used as the adhesive in a sandwich type reconstituted tobacco sheet.

TABLE V.—PROPERTIES OF BINDER FILM MADE BY ACID WASH PROCESS

Example	28	29	30
pH	7.1	7.1	7.1
Sheet wt., gm./ft.²	4.34	4.78	4.72
Neutralization agent	(1)	(2)	(3)
Integrator count	2864	3823	3268
Percent moisture	11.3	11.6	11.1
Percent elongation	1.08	1.60	1.26
Tensile, kg./in.	2.33	3.25	2.88
Tensile coefficient, kg./in.²/gm./ft.²	9.36	8.83	8.20
Work coefficient, gm. cm./sq. in.	4.88	4.88	4.05

(1) Conc. NH₄OH.
(2) 10% KOH.
(3) 10% NaOH.

In the table the terms have the following meanings:

(1) Integrator count is proportional to the area under the stress-strain curve recorded by an Instron Test Machine.

(2) Percent moisture—The percentage of moisture contained in the entire sheet (wet basis).

(3) Percent elongation—The percentage elongation, at breakage, on an Instron Test Machine.

(4) Tensile, kg./in.—The breaking strength of a 10 cm. test strip which is 1 inch wide; an average of 10 strips.

(5) Tensile coefficient—Kg. per gm. of basis weight.

(6) Work coefficient, gm. cm./sq. in.—Work coefficient is proportional to the product of the tensile strength and the elongation.

Example 31

The following ingredients were mixed into 900 parts of hot water and heated for 3 hours at 85°–90° C., maintaining a pH of 7.0 by adding small amounts of aqueous ammonia.

	Parts
Mixed manufacturing byproducts and scrap tobacco	87.9
Diammonium ortho phosphate (DAP)	5.0
Glycerin	5.0
Corn syrup	2.0
Potassium sorbate	0.14

The slurry was then refined in a one gallon capacity Waring Blender for 5 minutes. With this amount of refining, the fibrous portion of the composition was seen to be well dispersed and thoroughly interlocked when examined in a droplet at a 1:9 dilution. This examination was facilitated by the addition of a small amount of Congo red dye.

The composition was cast on stainless steel panels with an eight inch angle knife set for wet film thicknesses of 0.020 to 0.045 inch, and dried on a steam table.

The wet strength of the films was determined on a Scott Serigraph IP-2 using 1 inch width strips. The center of each strip was wet with distilled water on both sides and allowed to become thoroughly wet through (30 seconds or one minute) before actuating the tensile mechanism. These films showed the extraordinarily high wet film strength of 140 grams at a film weight of 9.5 grams per square foot. A high wet strength is highly desirable in the preparation of cigarette filler, and assures that the subsequent manufacturing performance of this material will exceed that of the finest grades of leaf tobacco. Such strength is uniquely obtainable in the presence of the liberated form of pectin described in this disclosure.

We claim:

1. A process for producing reconstituted tobacco sheet material which comprises contacting comminuted tobacco parts with a treating solution containing a reagent comprising ammonium orthophosphate so as to destroy the alkaline earth metal cross-links which hold the pectinaceous material and thus occasioning release of the pectins

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by the formation of a precipitate comprising an insoluble calcium or magnesium salt, and finally casting the tobacco and the in situ formed pectins to form a reconstituted sheet, the pectins serving as a binder for the treated tobacco parts in the final sheet.

References Cited

UNITED STATES PATENTS

1,634,879 7/1927 Naoji et al.

3,012,935	12/1961	Howard	131—17
3,069,734	11/1956	Bandel	131—17
3,121,433	2/1964	Plunkett et al.	131—140
3,120,233	2/1964	Batista et al.	131—143 X

MELVIN D. REIN, *Primary Examiner.*

U.S. Cl. X.R.

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582-521 C13

United States Patent Office

3,353,541
Patented Nov. 21, 1967

3,353,541

TOBACCO SHEET MATERIAL

John D. Hind and Robert B. Seligman, Richmond, Va., assignors to Philip Morris Incorporated, New York, N.Y., a corporation of Virginia

No Drawing. Filed June 16, 1966, Ser. No. 557,903
1 Claim. (Cl. 131—17)

ABSTRACT OF THE DISCLOSURE

This disclosure relates to a process for producing a binder composition for use in the manufacture of reconstituted tobacco. The binder is made from tobacco plant parts and involves the use of the naturally occurring tobacco pectins, which are obtained by a process in which ammonium acid phosphate is employed to treat the tobacco plant parts. The treatment involves the destruction of the alkaline earth metal cross-links of the tobacco pectins, the release of the resulting tobacco pectins by a washing action and the depositing of the released tobacco pectins on the treated plant parts.

This application is a continuation-in-part of application, Ser. No. 336,009, which was filed on Jan. 6, 1964, now abandoned, and which, in turn, is a continuation-in-part of application, Ser. No. 240,130, filed Nov. 26, 1962, now abandoned, and application, Ser. No. 169,995, filed Jan. 31, 1962, now abandoned.

This invention relates, generally, to tobacco compositions and methods of producing such compositions. More particularly, the invention relates to improved adhesive materials as binders for reconstituted tobacco compositions. In addition, the invention relates to methods for the production of novel adhesive compositions comprising tobacco pectins, to reconstituted tobacco containing tobacco pectins as binders, and to smoking compositions made therefrom.

During the production and processing of tobacco products, including aging, blending, sheet forming, cutting, drying, cooling, screening, shaping and packaging, considerable amounts of tobacco fines and tobacco dust are produced. It is known that such tobacco fines and dust can be combined with a binder to form a coherent sheet, which resembles leaf tobacco and which is commonly referred to as reconstituted tobacco. One method for making reconstituted tobacco of this general character is disclosed in United States Patent No. 2,734,510, wherein the tobacco fines and dust are applied to a binder made of carboxymethyl cellulose, carboxylmethyl hydroxyethyl cellulose or a suitable salt thereof. The binder, in such compositions, ranges from about 5% to about 50% of the weight of the tobacco employed. United States Patent No. 2,708,175, describes a binder for reconstituted tobacco which consists of a plant gum, principally of galactomannan. United States Patent 2,592,554 to Frankenburg describes, as binders for reconstituted tobacco, various

water-soluble polysaccharides, such as alginic and pectinic acids and their sodium and potassium salts, derived from plants other than tobacco; for example derived from citrus fruits. However, the addition of cellulosic binders further increases the amount of cellulosic material in the product and tends to create an acrid and bitter smoke, when the product is used to make cigarettes. The natural hydrophilic colloid gums such as guar gum, locust bean gum, algin and other commonly used materials, such as Irish moss, have additional disadvantages. These materials contain proteins and other materials not found in tobacco which add distinctive flavors of their own to tobacco products during smoking. Thus, Frankenburg, in describing the use of various water-soluble polysaccharides derived from plants other than tobacco, teaches that care should be exercised that they must be in a state of refinement. Frankenburg teaches that these materials should be free of extraneous matter containing compounds of nitrogen, particularly proteins, and compounds of sulfur, phosphorus and the halogens; i.e., compounds giving undesirable products of combustion or dry distillation. Such refining is often a very tedious and difficult operation.

The present invention makes possible the production of improved reconstituted tobacco by a method which is simpler and more effective than the methods previously employed. The present method does not require refining of the binder and is, therefore, more easily and efficiently employed than other methods for making binders and for making reconstituted tobacco. The reconstituted tobacco which is obtained in accordance with the present invention need not contain any additional cellulose or proteinaceous material which is foreign to tobacco, since the binder which is employed may be derived solely from tobacco, and may be produced in such a manner that it contains no materials other than those which naturally occur in tobacco. Thus, reconstituted tobacco produced in accordance with the invention, can be so formulated as to be similar in physical properties and chemical composition to natural tobacco.

The term pectic substances [1] will mean those substances which are found in many plant products, and which consist essentially of partially methylated galacturonic acids joined in long chains.

The pectic substances found in tobacco plants contain acetyl groups and differ considerably from commercially available pectins found in other plants, including sugar beet pectins and citrus and fruit pectins. Tobacco protopectins are uniquely insoluble in hot water as compared with protopectins from many other sources and comprise mainly water-insoluble pectins (protopectins) consisting of the calcium and magnesium salts of partially esterified and slightly acetylated polymers of galacturonic acid. The divalent calcium and/or magnesium atoms act as cross-links between acid chains, thus making the polymers water-insoluble. As an illustration, the structure of the

[1] Note: Unless otherwise specified, the term "pectins" will, for convenience, hereinafter be employed interchangeably with the term "pectic substances."

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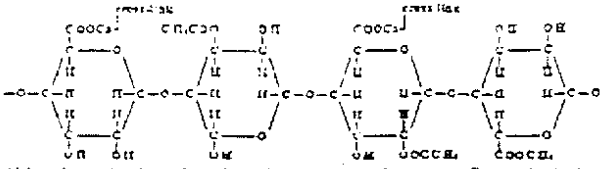
calcium salt of a polymer of galacturonic acid can be represented as follows:



Although pectins have long been known as constituents of plant tissue, it has been found extremely difficult to separate pectins from the remainder of plant compositions and to obtain them as homogeneous compositions. The recovery of pectins from tobacco is even more difficult than the recovery of pectins from other plants.

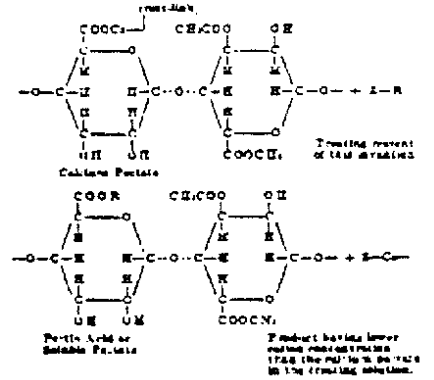
In accordance with the present invention, tobacco parts are bonded together by tobacco pectins which are specially prepared by a novel process which yields these pectins in a form in which they can be employed as binder materials. Our process for preparing tobacco pectins comprises first reacting tobacco parts, preferably in a form in which they present a large surface area, with an aqueous solution of a non-toxic reagent which is capable of reacting with and destroying the calcium and magnesium crosslinks in the pectinaceous substances which naturally occur in tobacco. After the calcium and magnesium crosslinks are destroyed, the tobacco pectins are liberated and made available for use as a binder. The tobacco pectins are then dissolved or dispersed in solution, or are at least sufficiently released from the interstices of the tobacco mass so that they form a coating on the surface thereof. Tobacco pectins which are dissolved or dispersed in the treating solution, are thereafter precipitated or deposited from the solution, so that they become available for use as a binder material. In this way, the tobacco parts can be bonded together by a binder material which is made of ingredients that are closely related to the naturally occurring ingredients of tobacco. The bonding can be accomplished without the need for the purification of the tobacco pectins, inasmuch as any materials which are present are closely related to the materials which are normally present in tobacco and, thus, do not add any undesired qualities to the tobacco.

The tobacco parts which can be employed in the present invention including tobacco leaves, stems and stalks, or a mixture of these, whether in sheet, flake, particulate or other form. Preferably, the parts are ground, cut or otherwise prepared in a form which presents a large surface area. The portions of the plant comprising the stems or midribs, and often referred to as tobacco petioles, are the preferred starting materials. Tobacco stalks contain lesser amounts of pectinaceous materials but can also be employed.

In the first step of the process of our invention, tobacco pectins are liberated from pectinaceous materials in tobacco by reacting the pectinaceous materials with a re- [illegible] the conditions of the reaction, is reactive with the calcium (and/or magnesium) contained in them to form a compound or product having a lower calcium ion, and, in the case of magnesium, magnesium ion concentration[1] in the treating solution than the naturally occurring calcium (or magnesium) pectate. This reagent may, for convenience, be hereinafter referred to as "cross-link destroying reagent."

The reaction may be generally represented by Equation I, which illustrates the reaction of one type of tobacco protopectin (a calcium salt of a polymer of galacturonic acid), wherein calcium cross links are present with the reagent of the present invention. In the equation,

R may be hydrogen, in which case the product is pectic acid, or R may be a monovalent inorganic cation, such as sodium, potassium or ammonium, in which case the product is a soluble pectate.



In one embodiment of this invention, the reagent, which can be, and preferably is, in aqueous solution, acts by forming a precipitate with the calcium or magnesium, in which case, it can be a water-soluble monovalent metal salt of the formula M_qX wherein M is a monovalent inorganic cation, q is an integer having a value of 1, 2, or 3 and X is an anion which may be monovalent or polyvalent, such that the calcium salt of the formula Ca_pX_q is essentially insoluble in the treating solution and p and q are integers corresponding to the functionality of X. Monovalent cations which are effective include the alkali metals such as sodium, potassium and lithium, and also include such monovalent cations as ammonium, and substituted ammonium ions $(NR_4)^+$, where R=aryl or alkyl. The anion portion of the molecule may be $CO_3^{=}$, $PO_4^{=}$, $HPO_4^{=}$, $H_2PO_4^{-}$, and the like. For example, the compound MnX could be sodium carbonate, Na_2CO_3, since sodium is a monovalent inorganic cation and calcium carbonate is essentially water insoluble. Additional representative examples of precipitating agents are the orthophosphates, metaphosphates and carbonates of sodium, potassium, lithium and ammonium. In the case of the orthophosphates, the anion portion of the molecule may be either $PO_4^{=}$, $HPO_4^{=}$, or $H_2PO_4^{-}$. Specifically, for example, when ammonium orthophosphate is used, the precipitate is calcium and/or magnesium ammonium phosphate. The pH of this reaction should be between about 5.8 and 10 and the temperature may be as high as 400° C. but should, preferably, be between about 25° C. and about 175° C. for a period of from about 1 minute to about 24 hours. Preferred precipitating agents which may be employed are the alkali metal carbonates, for example,

[1] By concentration we mean concentration or activity as set forth in Glasstone, "Textbook of Physical Chemistry," 2nd edition, p. 951, D. V. Nostrand Company, Inc.

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sodium carbonate and potassium carbonate. Particularly preferred precipitating agents which may be employed are the alkali metal phosphates and, more particularly, the alkali metal orthophosphates and the ammonium orthophosphates, such as the ammonium phosphates and ammonium orthophosphate, sodium orthophosphate, potassium orthophosphate, sodium dihydrogen orthophosphate, ammonium dihydrogen orthophosphate, potassium dihydrogen orthophosphate, diammonium monohydrogen orthophosphate, disodium monohydrogen orthophosphate and dipotassium monohydrogen orthophosphate.

In a second embodiment of this invention, the cross-link destroying reagent acts by sequestering the calcium or magnesium, thereby removing the calcium or magnesium atoms by forming a complex therewith. Suitable reagents of this type include any sequestering agent which will form a complex or chelate with the calcium and/or magnesium, thereby removing the calcium and/or magnesium and making them unavailable for recross-linking with the pectin. Illustrative of such sequestering agents are ethylene-diaminetetraacetic acid and similar amino acids, alkali metal polymetaphosphates such as tetra-metaphosphates, hexametaphosphates and trimetaphosphates, pyrophosphates and tripolyphosphates, such as sodium hexametaphosphate, tetrasodium pyrophosphate and pentasodium tripolyphosphate. The mechanism which occurs when a sequestering agent is employed is the formation of a chelate; calcium and magnesium ions are no longer available to combine with the pectate ions in solutions. Many naturally occurring amines and peptides are also effective as sequestering agents for calcium and/or magnesium. Representative examples include alanine, tartaric acid, glycine, glycyl glycine, glutamic acid, serine, tyrosine and di-iodo-l-tyrosine. Amino acids that are effective as chelating solubilizing agents include beta alanine, N,N-diacetic acid; amino barbituric acid, N,N-diacetic acid; 2-amino-benzoic acid, N,N-diacetic acid; beta-aminoethylphosphonic acid, N,N-diacetic acid; beta-aminoethylsulfinic acid, N,N-diacetic acid and ethylene-diamine-tetraacetic acid. The pH of this reaction should, preferably, be between about 4 and about 10 and the temperature should, preferably be between about 0° C. and about 145° C. for a period of from about 1 minute to about 24 hours.

A cross-link destroying reagent may also function partially as a precipitating reagent, in accordance with the first embodiment of this invention, and partially as a sequestering agent, in accordance with the second embodiment of this invention. Such a reagent, for example, is diammonium monohydrogen orthophosphate (DAP), which is a particularly preferred material, in accordance with the invention.

In a third embodiment of this invention, the cross-link destroying reagent is an acid wash which forms the released but insoluble free pectic acid and soluble calcium and magnesium salts. Generally the acid wash will comprise an inorganic acid, such as hydrochloric acid, phosphoric acid, sulfuric acid or a similar acid, which will form soluble calcium and magnesium salts under the following conditions. Hydrochloric acid and sulfuric acid are particularly preferred. The acid may be employed as 0.25 N to [illegible] solutions, but is preferably employed as 0.5 N to 1.0 N. The exact dilution and amount to be employed will depend with the particular acid which is used, it only being necessary that sufficient acid be present to convert the calcium and magnesium present in the tobacco being treated to the calcium and magnesium salts of the acid. The acid treatment is preferably conducted at a temperature of from about −1° C. to about 50° C. The acid treatment comprises reacting the tobacco parts with the acid until the resulting mixture has a pH of from about 1.0 to about 2.5. Preferably, the pH is brought to from about 1.0 to about 1.7, the most desirable pH being between 1.15 and 1.55. This treatment will generally be conducted from about 10 minutes to 24 hours, depending in part on the size of the tobacco particles. The acid conditions which are necessary for this embodiment of the invention may be achieved by the use of ion exchange resins which may be used, with suitable recycle to obtain the desired pH of the solution during treatment. The ion exchange resins may be used and regenerated in accordance with the usual practices for such resins.

Preferably, the mixture resulting from the acid treatment is then washed with water. This water wash step is preferably conducted at a temperature of from about 15° to about 35° C. and, preferably, distilled water is employed. When this wash step is employed, sufficient water should be used to remove the calcium and magnesium salts of the acids, which salts are formed in the above-described treatment; thus, there should be at least 2 volumes of water per volume of the mixture resulting from the acid treatment. The wash water is separated from the tobacco by any suitable means, for example, by conducting the wash in a centrifuge, filter press, Buchner funnel, or any other apparatus from which liquids can be substantially removed from solid material.

In summary, in the first step of the first embodiment of this invention the treating agent Z-R attacks the calcium and/or magnesium cross-links of tobacco protopectin and forms a precipitate which is a salt of calcium and/or magnesium, thus removing the calcium and/or magnesium from the protopectin and from the solution. In the first step of the second embodiment, the treating agent Z-R is a sequestering agent which forms a chelate of the magnesium and/or calcium from the tobacco protopectin and makes the calcium and/or magnesium unavailable for recombining with the pectins. With certain reagents, such as DAP, the first step of the present process may comprise a combination of the mechanisms of the first embodiment of this invention and the mechanism of the second embodiment of this invention. In the first step of the third embodiment of this invention, the treating agent Z-R is an acid which attacks the calcium and/or magnesium cross-links of the tobacco protopectin and forms the soluble calcium and/or magnesium salts, which are then washed away from contact with the pectins.

In the first step of each of the first two embodiments of the invention, the pectin which results is in condition for release from the tobacco cell structure. R in Equation I being a monovalent inorganic cation such as sodium. In the first step of the third embodiment, the insoluble pectic acid resulting from the acid treatment must be reacted with an alkaline material before it is in condition for release from the tobacco cell structure.

The acid treated pectins are placed in condition for release by bringing the mixture resulting from the acid treatment, and preferably, after the water wash described above, to a pH of from about 5.0 to about 10.5 and, preferably, from about 6.3 to about 8.5, by the addition of an alkaline material. Suitable alkaline materials include ammonium hydroxide and alkali metal hydroxides, for example, sodium hydroxide, potassium hydroxide and lithium hydroxide, and alkali metal salts, such as sodium bicarbonate, sodium carbonate, sodium phosphate, and similar salts to convert the pectic acid to a soluble form. The alkaline material may be any water-soluble compound containing a monovalent inorganic cation and capable of producing hydroxide ions when dissolved in water. The temperature of this step may be from about −1° C. to about 45° C., but is, preferably, from about 15 to about 35° C. The alkaline material is preferably in the form of solid particles or in the form of a solution having a concentration of from about 5 to about 50%.

Once the tobacco pectins have been liberated from the tobacco, by the removal of the calcium and magnesium cross-links, they should be released from the interstices of the tobacco. That is, they will be made available to the

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solution or suspension or, in certain instances, they will be merely deposited on the surface of the tobacco particles. This comprises the second step of the process of the present invention. In embodiments 1 and 2 of the invention, this release or second step may be accomplished concurrently with the first step by reacting with the solution of the treating reagent. In embodiment 3, however, as indicated above, the insoluble pectic acid resulting from the treatment should be reacted with an alkaline material before it can be released. In such event, the release may be concurrent with the addition of the alkaline material due to a washing action. In any case, additional treating liquid or water may be used to effect the release through a washing action of the treated tobacco particles.

In accordance with the third step of our process, the liberated and separated tobacco pectins can next be precipitated or deposited in a relatively free form (as compared with the tobacco pectins as they were originally present in the tobacco), from the treating solution, for example, by being formed into the insoluble pectic acid or into an insoluble salt of pectic acid or by the action of a water-miscible solvent, such as acetone or ethyl alcohol to cause a water-soluble salt of pectic acid to go out of solution. In the case of tobacco pectin solutions such as solutions of sodium and potassium pectinates and/or pectates, this can be accomplished by acidifying the solution until the pectins precipitate or by adding a gelation agent, such as an alcoholic solution, preferably having a pH of from about 1 to 9 and, preferably, from about 1 to 5. The pH of the alcoholic solution can be regulated by the addition of a mineral acid, such as HCl, to the alcohol. Although the preferred gelation agent is ethanol, any water-miscible organic solvent having up to about 10 carbon atoms may be employed, for example, a ketone, such as acetone, or a diether, such as dioxane. Water-immiscible solvents such as ether, for example, ethyl ether, can be used, if combined with a water-miscible solvent, such as acetone.

The tobacco pectins can be recovered by concentrating the solution or suspension in which they are present until they precipitate. This precipitate might also be characterized as an extractable mass, since the pectin solution, upon concentration, generally becomes progressively more viscous until it finally dries to leave a deposit in a glassy solid state.

While the tobacco pectins can be separated and purified before use as a binder in reconstituted tobacco sheets, they are preferably employed just as they are produced in situ, i.e., in combination with the treated tobacco plant parts from which they were obtained, the entire combination comprising the binder for reconstituted tobacco sheets or, under some circumstances, the entire combination comprising essentially the entire components of a reconstituted tobacco sheet. By using the entire mixture, no original tobacco flavors are lost, all of the tobacco is employed, and no expensive and time-consuming refining operations are required.

Although it is not necessary, the thixotropic properties of solutions containing soluble pectins can be adjusted in the preparation of a cured sheet by the addition of such materials as calcium chlorides. If any complex or precipitate formed in the first step of this process is present with the soluble pectates, the thixotropic properties of the mixture can also be adjusted by adjusting the pH to precipitate calcium and magnesium pectates.

A preferred preliminary step, in accordance with the present invention, comprises washing the tobacco plant parts, which are preferably ground or cut to a relatively small size, with cold water. This water wash serves to remove impurities which might otherwise hinder the subsequent treatments in accordance with the present invention. It is particularly desirable to employ such a cold water washing step when alkali metal carbonates are employed as the reagent in the first step. Generally, sufficient water should be used during such a water wash

operation to cover all of the tobacco plant parts present. The temperature of the water may be between —1° C. and 100° C., but is preferably about 20° C., and the water wash should generally continue for a period of from about ½ to 2½ hours. Agitation during the wash is desirable, but not necessary. After the water wash has been completed, the water can be removed from the tobacco parts by filtration, decantation or other suitable means.

As discussed above, a particularly preferred embodiment of the present invention involves the use of an ammonium or alkali metal orthophosphate, such as diammonium monohydrogen orthophosphate (DAP), for the release of the tobacco pectins. The DAP will, generally, be added to the tobacco plant parts, which may, for example, be bright tobacco parts, burley tobacco parts, or a mixture of the same, in an aqueous solution. The concentration of the DAP in the aqueous solution is not critical, but will, generally, be in the range of 0.5–5.0% by weight. The DAP and water may be added separately to the tobacco. The amount of DAP should, preferably, comprise from about 0.01 to about 0.5 part, and, most preferably, from about 0.05 to 0.35 part (by weight) per part of tobacco being contacted. A humectant, such as glycerin or triethylene glycol, may be present, if desired, at about 0.5 to 1 part per part of DAP. The temperature during the DAP treatment of the tobacco may vary between room temperature and about 150° F. or higher, depending on the type of tobacco being treated. Under pressure, even higher temperatures may be employed. The pH of the mixture is, preferably, maintained at a value about 7.1 to 9.0, which may conveniently be accomplished by the addition to the solution of concentrated aqueous ammonia. By agitating or stirring such a mixture under the above-described conditions for from about 1 minute to about 1 day, and preferably, from about 1 hour to about 5 hours, the tobacco pectins are liberated, released and deposited in the tobacco plant parts to form a binder composition. The resulting mixture may then be refined, for example, in a disk refiner, until substantially all the pulp (in excess of the water present), can be shaken through a screen of approximately 18 mesh to produce a binder composition which is ready for use in the manufacture of reconstituted tobacco sheets.

The term "tobacco pectins" as used throughout this specification means "liberated tobacco pectins" and comprehends pectins which have been freed or liberated from tobacco and are, therefore, not bound into the tobacco structure, as differentiated from the insoluble, naturally-occurring protopectins which are bound into a plant cell structure. The term includes the free pectinic or pectic acid, as well as soluble salts such as the sodium, potassium, ammonium, pectates and pectinates, and insoluble salts such as the calcium and magnesium pectates and pectinates depending on what method is employed to liberate and obtain them from the naturally occurring insoluble protopectins.

The tobacco pectins produced or liberated in situ or isolated by means of this invention can be used as the sole binder material for reconstituted tobacco, i.e., no other materials need be added to make the sheet. They can be sprayed, extruded or cast, thus facilitating application onto a moving belt carrying tobacco dust. Under proper conditions of formulation and processing, reconstituted tobacco made with the tobacco pectins produced by this invention exhibit excellent physical and aromatic properties. The ultimate tensile and wet strengths of the reconstituted tobacco are good. While no other materials need be added to the pectinaceous binder, other materials can be added, if desired. For example, organic acids and preservatives which may in themselves be of tobacco origin may be added. Plasticizers, such as glycols and polyglycols, and humectants, such as glycerin, may also be added, if desired. In addition, the gel strength of the tobacco pectins can be regulated by partial precipitation

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to control such rheological properties as viscosity, fluidity and elasticity. Other additives or dispersants may be added in small amounts to regulate slurry qualities, provided, however, that such substances are not added in large enough quantities to adversely affect the flavor or aroma of the final product. Furthermore, the tobacco pectins can be combined with water-soluble gums or water dispersible gums commonly used as binders for tobacco sheets such as methyl cellulose, sodium carboxymethyl cellulose, guar gum, locust bean gum, or alginates, although it is preferred to minimize or eliminate such additions in order to obtain a product which most closely resembles natural tobacco.

The product from treating the tobacco plant parts in accordance with the methods of the present invention may be cast directly and dried and cut into particulate material similar in physical form to ordinary smoking tobacco and so used, preferably mixed with tobacco leaf cut or shredded in the usual manner. The product may be cast in sheet form, in blocks or as threads or other shapes, as desired. An important use, however, of the prepared comminuted slurry or easily molded isolated pectinaceous mass is as a binder for ground tobacco and for the making of corresponding tobacco products suitable for smoking. Sheet material of widely different properties may be formed by suitable variations in the manner of forming. One method and product comprises flowing the comminuted slurry onto a moving belt and applying a layer of dry ground or fragmented tobacco to the wet adhesive surface. If desired, there may be first applied to the belt a layer of the tobacco, followed by a layer of the binder, and then a top layer of the tobacco. Various additives may be included with the ground tobacco such as flavorants, plasticizers and aromatic substances. The web is ultimately dried and then suitably moistened and rolled up. Such methods of forming continuous sheets are known generally in the art and the details need not be further described. Representative of this procedure is the apparatus and method disclosed in U.S. Patent 2,734,513.

Another method of forming a reconstituted tobacco product, with the slurry of the isolated tobacco pectins as a binder, comprises mixing ground tobacco thoroughly therewith into a mass of dough-like consistency and then casting the mass in sheet form onto a moving belt surface followed by drying and reconditioning in accordance with well known procedures. Representative of this procedure is the apparatus and method disclosed in U.S. Patents 2,708,-927 and 2,769,734. Obviously, the reconstituted tobacco may also be formed by molding or other suitable means.

A particularly preferred aspect of the present invention comprises employing, as a binder or directly, the mixture of tobacco and tobacco pectins which have been produced in situ, without any separation steps and without the necessity for any additional adhesive materials.

The following examples are illustrative:

Example 1

Tobacco stems (10 parts) were covered with cold water and leached for ½ hour. The water was then decanted and discarded. A treating solution, made from 1 part of sodium carbonate dissolved in 60 parts of water, was added to the leached parts. This mixture was heated at its boiling point for 30 minutes at atmospheric pressure and then for 20 minutes at 20 p.s.i.g. In the course of this treatment, the tobacco pectins were dissolved from the tobacco parts. The entire wet mass (pulp mixture) was [illegible] and ground in a Waring Blendor so that it would [illegible] through a 50 mesh screen. The resultant material had [illegible] properties and was thixotropic in nature.

To [illegible] material was added 2 parts of glycerin, to serve as [illegible] plasticizer. The pH of the resulting mixture was adjusted [illegible] by the addition of a solution of 10% hydrochloric acid. [illegible] gram of sodium carboxymethyl cellulose (CMC) was [illegible] the mixture, giving a proportion of [illegible] parts of CMC to 10 parts of total solids in the mixture. Since it was desired to use this mixture as a sprayed tobacco binder, the CMC was employed in order to adjust the spraying qualities. The mixture of (a) treated tobacco plant parts, including the liberated tobacco pectins, (b) the sodium carboxymethyl cellulose and (c) the glycerin had a viscosity which was suitable for spray application of the mixture as a binding material for reconstituted tobacco. This material was sprayed on top of an undercoating of tobacco dust, of an 80 mesh size, which had been dusted on a wet belt. Another coat of the tobacco dust was laid on top of the binder. In general an apparatus similar to that described in U.S. Patent 2,734,513 was employed. As in said patent, the reconstituted sheet was dried and was then humidified to the desired moisture content. The control in Table I was made in a conventional manner, using a relatively large amount of CMC in proportion to the pulped washed tobacco stems. The test data as observed are recorded in Table I.

TABLE I

Sample	Sheet 1	Sheet 2	Sheet 3	Control
Part CMC [1]	1/10	1/10	1/20	1/1.4
Stiffness (grams) [2]	12.5	12.1	12.7	11.6
Tensile, lbs./in.²	.80	.86	.88	1.1
Tensile, kg./cm.²	.71	.78	.83	.74
Elongation (percent) [3]	8.2	7.7	8.0	3.9
Burning Rate, mg./sec. [4]	1.8	1.7	1.9	1.7

[1] Part CMC per parts of total solids (pulped tobacco stems) in the slurry (including [illegible] pectins).
[2] The percentage of moisture employed in the various sheet test [illegible].
[3] The breaking strength of a 10 mm. test strip when in 1 inch grips [illegible] a rate of 10 in./min.
[4] Cigarette [illegible] was made employing the application of a 450 grams weight for 30 seconds.
[5] The general description, as found [illegible] Test Standard.
[6] Free (flaming) burning rate of a single test strip, in air.

The reconstituted tobacco sheets made pursuant to this example were shredded and made into cigarettes. The test cigarettes, as well as control cigarettes made from standard reconstituted tobacco sheet, were submitted to a smoking panel for subjective testing. The panel found that the smoke of the cigarettes made by the method of this invention was significantly less harsh than the smoke obtained from the control cigarettes. A pleasing vanillin odor was found to be transmitted into the aerosol phase of the smoke from the test cigarettes made employing reconstituted tobacco made by the present invention.

Example 2

Fifty grams by weight of burley tobacco stems were thoroughly washed in about 5 liters of cold water for three hours. The washed tobacco stems were then mixed with 500 grams of water having dissolved therein 5 grams of sodium carbonate. The resultant mixture, containing about 8% solids, was steamed under atmospheric pressure for 30 minutes and under a pressure of 20 p.s.i.g. for an additional 20 minutes. At the end of this time, the mixture was allowed to cool and the liquid was separated from the solid materials. The solid materials were treated in a cider press to recover as much as possible of the remaining liquid. The liquid was added to a previously prepared coagulant bath which consisted of ethanol and hydrochloric acid, in an amount to adjust the pH of the coagulant to about 1.0. The resulting mixture (which had a pH of about 1.0) was alternately stirred and allowed to settle for a period of two hours. At the end of this time the mixture was strained through a cloth sieve and the filtrate was discarded leaving a solid, gel-like mass, consisting essentially of pectinaceous materials combined with approximately 10 parts of liquor. The yield of pectinaceous materials was 15%, based upon the dry weight of the starting tobacco plant parts. The mass was observed

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to be thixotropic, soluble in water at a pH of about 6 and soluble in a sodium carbonate solution.

The tobacco pectins thus isolated were quite impure and had a color characteristic of tobacco. The solid mass was then redissolved in a sodium carbonate solution and the resultant solution was poured into an acidified ethanol bath similar to the coagulant bath used earlier. The recoagulated solid was obtained by filtration and dried in an oven at 105° C. The dried tobacco pectins were in the form of an opaque, substantially colorless sheet. When the sheet was pulverized, a white powder was obtained having a distinct and pleasing odor similar to vanillin.

Ten grams of the dried tobacco pectins prepared above were swelled in 100 ml. of cold water. The mixture was then added to a slurry consisting of 8.5 grams of refined kraft pulp dispersed in 200 ml. of water. The resultant mixture was homogenized in a Waring Blendor. Four grams of glycerin were then added to the mixture to serve as a humectant. The resulting mixture was cast into a binder film, using a Gardiner casting knife set to produce a sheet having a wet thickness of 30 mils. The physical properties of this sheet, after drying, were tested and were found comparable to the properties of conventional binder films. The physical test data are given in Table II. The control material was made by mixing pulp, which had been refined with NaOH, with sodium carboxymethyl cellulose (CMC) and a humectant, as is shown in Table II.

TABLE II

	Test Binder-Film	Control Binder-Film
Basis Weight	(a)	(b)
Basis Weight, gms./M.²	5.3	5.81
Caliper, Percent	16.3	14.9
Density, Gm./cc.	0.88	0.87
Instron Tensile, psi.	5.3	5.3
Elongation, Percent	4.1	4.1
Work to Rupture, gram cm./cm. in.²	87.83	81.9

[a] Dried tobacco pectins precipitated from tobacco pectates.
[b] CMC—Pulp—CMC (sodium carboxymethyl cellulose) and pulp, homogenized by Waring Blendor and containing about 20% CMC.
[c] Tensile strength is proportional to the product of the tensile strength and the elongation.

Example 3

One hundred grams of burley tobacco stems were covered with distilled water, leached for 30 minutes and then drained. The washed stems were then mixed with a solution containing 10 grams of diammonium monohydrogen orthophosphate in 600 ml. of water. The resultant slurry was heated for 1 hour at a temperature of from 90 to 100° C. The pH of the reaction mixture, after completion of the reaction, was about 7. The entire reaction mixture was homogenized in a Waring Blendor. The solids content was determined to be 5% by weight.

Two grams of glycerin, to serve as a humectant, were blended with 200 grams of the above reaction mixture. A film of 30 mil wet-thickness was cast from this mixture. The extruded film was found to have the property of being able to slide off a surface even when newly cast. After the film was partially dried, it was found that it could be peeled off a plate by hand, while still partially wet, and then hung up to dry like wet cloth. This property offers definite advantages in the manufacture of tobacco products.

Physical tests were carried out on the dried film thus prepared and were also carried out on a control film made by using conventionally pulped tobacco plant parts with sodium carboxymethyl cellulose (CMC) as the binder. A film of binder as made in this example was burned and was found to give a pleasant aromatic smoke. The test data are given in Table III.

TABLE III

	Test Binder-Film	Control Binder-Film
Basis Weight	(a)	(b)
Basis Weight, gms./M.²	5.9	5.9
Caliper, Percent	14.3	14.3
Density, Gm./cc.	0.83	0.87
Instron Tensile, psi.	5.6	5.3
Elongation, Percent	5.1	4.1
Work to Rupture, gm. cm./cm. in.²	51.0	51.9

[a] Based on total nitration.
[b] Test was sodium gel formed in situ.
[c] CMC—Pulp.
[d] Gm. per sq. cm. of basis weight.
[e] Gm./in. for a 1/2 cm. test strip.
[f] Work to rupture is proportional to the product of the tensile strength and the elongation.

Examples 4–10

In each of these examples, burley tobacco stems were covered with distilled water, leached for 30 minutes, and drained. One hundred grams of stems were treated with the agent indicated in Table IV. The agent was dissolved in 600 ml. of distilled water in an amount sufficient to make up a 10% (by weight) solution. The leached tobacco stems were placed in said solution and heated in a steam bath for 1 hour at a temperature of from 90 to 100° C. (Except for the example in which Versene was employed. That example was conducted at room temperature.) A five ml. aliquot was taken from the resulting mixture and mixed with 20 mls. of ethyl alcohol in a graduated cylinder whereby a precipitate of tobacco pectins was obtained. The amount of precipitate in the graduated cylinder was observed. Each of these precipitates of tobacco pectins was suitable for use in the preparation of a reconstituted tobacco sheet in accordance with the methods taught by the present invention. The results obtained in these experiments are presented in Table IV below:

TABLE IV

Reagent:	Volume of coagulated tobacco pectins
Pentasodium tripolyphosphate	15
Sodium hexametaphosphate	5
Diammonium monohydrogen orthophosphate (DAP)	15
Trisodium orthophosphate	15
Disodium monohydrogen orthophosphate	15
Dihydrogen monosodium orthophosphate	5
Versene (tetrasodium salt of ethylenediaminetetraacetic acid)	15

Example 11

One hundred grams of burley tobacco stems were covered with distilled water, leached for 30 minutes and drained. A solution of 10 grams of Versene (tetrasodium salt of ethylenediamine-tetraacetic acid) in 600 ml. of water was added to the stems and the mixture was made basic with aqueous sodium hydroxide. The resulting mixture was then heated for 1 hour at room temperature. The entire mixture (a pulp) was homogenized in a Waring Blendor and the solids content was determined to be 5% by weight. A 200 gram portion of the pulp, thus prepared, was combined with 2 grams of glycerin, as a humectant, and then cast into a film of 30 mils thickness by the use of a Gardiner casting knife. The resultant sheet was found to have satisfactory physical properties and, upon burning, exhibited a very pleasing aroma.

Example 12

Green bright tobacco leaves were soaked in isopropanol to remove sugars and chlorophyll. The midrib was stripped out of the leaves leaving the web. One thousand grams of the web (82 grams on a solids basis) were placed in

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a porcelain bucket and covered with boiling distilled water containing 9.3 grams of diammonium monohydrogen orthophosphate (DAP). The DAP was added on the basis of 10 grams of DAP per 100 grams of stems (which contained 12% moisture, by weight). The resulting mixture was boiled at low heat for one hour. The juice was then first expressed from the mixture by hand. Solids, for example cellulose and sand, were then further separated from the juice by centrifuging. The clear juice from both operations was then mixed with 70% ethanol to form a gel, which was then squeezed out in cheese cloth. The gel was transferred to a Buchner funnel where it was washed first with acetone and then with ethyl ether. Finally, the gel was placed in a vacuum desiccator and dried. The gel was suitable for use in the preparation of a reconstituted tobacco in accordance with the teachings of the present invention.

Example 13

Burley tobacco stalks were separated into cortical tissue, woody tissue, and pith. A sample of each was steam cooked with 10% sodium carbonate for 30 minutes at atmosphere pressure and for 20 minutes at 20 p.s.i.g. After this cooking, the woody tissue was still hard and could not be pulped in a Waring Blendor. The cortical tissue and pith were soft and pulpable. Each of these latter two preparations were suitable for use in the preparation of reconstituted tobacco.

Example 14

Coarse ground bright stem tobacco fines (30 grams) were washed thoroughly in cold water, and then placed in boiling aqueous solution of 3 grams of diammonium monohydrogen orthophosphate and cooked for 5 minutes. The mixture was then placed in a Waring Blendor. After a very short period in the blendor, the mixture was converted to a viscous, fine, impalpable slurry; wherein the tobacco stem particles had been completely separated to units of cellular size. This impalpable mass was suitable for use as a binder in reconstituted tobacco. When rinsed with some of the cold water washings removed in the first step, the impalpable mass immediately became viscous and eventually jelled to a soft mass.

For further comparison, a similar sample of coarse ground bright stem fines (30 grams), was dispersed in boiling water and cooked for about 30 minutes with three grams of diammonium monohydrogen orthophosphate. The pH of this mixture was brought to a value of 7.1 by the addition to the mixture of 30% aqueous ammonia. The granules of tobacco could then be refined to a palpable pulp, similar to the soft mass produced in the experiment described in the first part of this example.

Example 15

Burley tobacco stems (¼ pound) were covered with distilled water, allowed to stand several hours and the water decanted. This step was repeated several times and finally the stems were covered with distilled water containing 50 ml. of concentrated HCl and left overnight at 24° C.

After standing overnight the acidic water was decanted and the stems washed repeatedly free of HCl until the wash water gave no precipitate when treated with silver nitrate solution. The stems were then covered with distilled water containing 15 grams of sodium carbonate and left overnight at 24° C. The pH of the sample, the following morning, was 8.3. The stems were swollen and soft, were easily disintegrated with the fingers and the mixture homogenized and employed, as a binder [illegible]

Example 16

The [illegible] in this [illegible] was large scale equipment, [illegible] 200 gallon conical bottom, open top, stainless steel tank, fitted with a Cowles high shear mixer.

One hundred and forty-eight gallons of water were placed in the tank and heated to a temperature of 201° F. One hundred and twenty-eight pounds of bright tobacco (milled to pass a 6 mesh per inch sieve) stems were added, while operating the Cowles mixer at a low speed. Almost immediately after the addition of the tobacco, nine and one-quarter pounds of diammonium monohydrogen orthophosphate (technical grade) were added to the mixture. Ammonia (assaying 28%, by weight NH_3) was added to adjust the pH to 7.1. The mixing speed was increased to 1700 r.p.m. After a period of 3 minutes the temperature of the mixture was 194° F. Most of the particles in the mixture were soft enough to be smeared by hand and the mixture had a jelly-like consistency. The mixing was continued for 1 hour to obtain the highest possible state of disintegration, although a 15 minute period appeared to be sufficient for this purpose.

The viscosity of the mixture was found to be 10,400 cps. and its solids content was found to be 8.05% by weight.

This mixture was then employed as a binder, being pumped through a filter to storage tanks and subsequently sprayed on tobacco by the method and equipment described in U.S. Patent 2,734,513. It was applied at the rate of 3 grams per square foot to form a reconstituted tobacco sheet having the following physical properties:

Basis weight, gms./ft.² (1)	10.2
Moisture content (percent by weight)	13.0
Tensile, kg./in.	0.82
Fold tensile, kg./in.	0.62
Work-to-break, gm. cm./in.² (2)	16.0

(1) Weight of all the ingredients, including tobacco pulp, essentially free of moisture.
(2) Compared by an integrator attached to the Instron Tensile Tester.

Example 17

Burley tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then dried and ground and used to make a binder as follows:

One hundred parts of water were brought to a temperature of 195° C., and to this were added:

7.00 parts by weight (dry basis) of the washed burley stems,
1.05 parts diammonium phosphate, and
0.70 part of glycerin, as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 7.1 but no higher than 9.0.

The mixture was then stirred for one hour and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be thrown through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 18

Burley tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then used directly in the wet condition to make a binder as follows:

One hundred parts of water were brought to a temperature of 195° C., and to this were added:

7.00 parts by weight (dry basis) of the washed burley stems,
1.05 parts diammonium phosphate, and
0.70 part of a glycerin, as a humectant.

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Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 7.1 but no higher than 9.0.

The mixture was then stirred for one hour and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 19

Burley tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then dried and ground and used to make a binder as follows:

One hundred parts of water were brought to a temperature of 195° C., and to this were added:

7.00 parts by weight (dry basis) of the washed burley stems,
1.05 parts diammonium phosphate, and
0.70 part of triethylene glycol (TEG), as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 7.1 but no higher than 9.0.

The mixture was then stirred for one hour and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 20

Burley tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then used directly in the wet condition to make a binder as follows:

One hundred parts of water were brought to a temperature of 195° C., and to this were added:

7.00 parts by weight (dry basis) of the washed burley stems,
1.05 parts diammonium phosphate, and
0.70 part of triethylene glycol (TEG), as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 7.1 but no higher than 9.0.

The mixture was then stirred for one hour and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 21

Bright tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then dried and ground and used to make a binder as follows:

One hundred parts of water were brought to a temperature of 100° C., and to this were added:

7.00 parts by weight (dry basis) of the washed bright (flue cured) stems,
1.05 parts diammonium phosphate, and
0.70 part of glycerin, as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 8.0 but no higher than 8.5.

The mixture was then stirred for four hours and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 22

Bright tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then used directly in the wet condition to make a binder as follows:

One hundred parts of water were brought to a temperature of 100° C., and to this were added:

7.00 parts by weight (dry basis) of the washed bright (flue cured) stems,
1.05 parts diammonium phosphate, and
1.05 parts diammonium phosphate, and
0.70 part of glycerin, as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 8.0 but no higher than 8.5.

The mixture was then stirred for four hours and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 23

Bright tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then dried and ground and used to make a binder as follows:

One hundred parts of water were brought to a temperature of 100° C., and to this were added:

7.00 parts by weight (dry basis) of the washed bright (flue cured) stems,
1.05 parts diammonium phosphate, and
0.70 part of triethylene glycol (TEG), as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 8.0 but no higher than 8.5.

The mixture was then stirred for four hours and subsequently refined in a disk type refiner until better than 99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 24

Bright tobacco stems were washed in cold water whereby, from about 75 to 80% of the natural content of water-soluble substances were removed in the wash water. The stems were then used directly in the wet condition to make a binder as follows:

One hundred parts of water were brought to a temperature of 100° C., and to this were added:

7.00 parts by weight (dry basis) of the washed bright (flue cured) stems,
1.05 parts diammonium phosphate, and
0.70 part of triethylene glycol (TEG), as a humectant.

Concentrated aqueous ammonia was then added to bring the pH of the mixture to a value of at least 8.0 but no higher than 8.5.

The mixture was then stirred for four hours and subsequently refined in a disk type refiner until better than

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99% of the pulp (in excess water) could be shaken through an 18 mesh sieve.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 25

In the acid wash treatment of tobacco parts for removal of alkaline earth minerals of the tobacco pectin, it is necessary to use quite large volumes of water if it is desired to reduce the soluble anion content of the product to a very low level. In this case, the use of a cation exchange resin in a closed loop system with the tobacco parts can make it possible to conduct the process with limited amounts of water and acid.

The use of a resin (such as Dowex 50W) in this way does not at all alter the principles of this form of treatment since the resin merely serves as a convenient reservoir of acidity, continuously reconditioning effluent from the stems for reuse in the extraction.

In the following example, the use of a cation exchange resin permits the treatment of stems with a small fraction of their natural nitrate ion content. Hydrochloric acid is used at intervals to regenerate the resin, but never directly contacts the tobacco.

The apparatus used for ion-exchange extraction of calcium from tobacco stems consisted of a 20 inch diameter washing column fitted with a 20 mesh screen to support the charge of stems and a 6 inch diameter Pyrex column filled with 13 pounds of Dowex 50W-8 acid-form ion exchange resin beads. The valves and piping permitted a pump to be used either for recycling waters from the stem charge through the column or for separately regenerating the resin with hydrochloric acid solution.

In operation, fifty pounds of stems were placed in the washing column and rinsed with cold water until they were nearly free of water-soluble material. Then, the effluent waters were diverted through the ion exchange bed, returning by gravity to the top of stem charge. When the aqueous extract from one pound of burley stems was added to the recycling waters, a pH of between 1.5 and 2.0 was soon attained. During the treatment, the waters flowing from the resin column were monitored for their calcium content. (An appreciable precipitate from 30 ml. of resin effluent treated with ammonium oxalate and made basic with ammonia indicated that the resin was saturated with calcium and needed to be regenerated with hydrochloric acid.) After sixteen hours of operation, and one regeneration of the ion exchange resin, the treatment was considered complete. A subsequent analysis showed that the calcium content of the stems had been reduced to less than one-sixth of the starting value of 4.5% CaO. The stems were rinsed twice with cold distilled water, pressed lightly, and redried in a tobacco processing oven to a final moisture content of about 4%, and ground to pass a 40 mesh screen. When a small sample of this powder was stirred with a little cold ammonia, it dispersed immediately, forming a stiff paste.

The resulting material was then employed as a binder for tobacco plant parts to form a reconstituted tobacco sheet in a manner similar to that described in Example 16.

Example 26

Twenty grams of tobacco fines were washed in 1 liter of distilled water to get rid of the tobacco solubles. The fines were then admixed with 1 N hydrochloric acid until the mixture had a pH of 1.35. The pH was checked on a Beckman pH machine and the curves were also plotted against a titration of distilled water.

After titration, the acid was washed off the fines with three 400 ml. portions of distilled water. The fines were redisbursed in 400 ml. of distilled water and sodium bicarbonate was added to bring the pH to 7. The slurry was cast at a thickness of 20–50 mils and dried to form a sheet and the resulting sheet was used as the adhesive binder in sandwich type reconstituted tobacco sheet.

Example 27

Twenty grams of tobacco fines were washed in 1 liter of distilled water to get rid of the tobacco solubles. The fines were then admixed with 1 N hydrochloric acid until the mixture had a pH of 1.35. The pH was checked on a Beckman pH machine, and the curves were also plotted against a titration of distilled water.

After titration, the acid was washed off the fines with a 400 ml. portion of distilled water. The fines were redisbursed in 400 ml. of distilled water and sodium bicarbonate was added to bring the pH to 7. The slurry thickened to a viscous mass, which is evidence that the pectin had been solubilized. The slurry was cast at a thickness of 30–50 mils and dried to form a sheet and the resulting sheet was used as the adhesive binder in sandwich type reconstituted tobacco sheet.

Examples 28–30

In each of these three examples, 20 gms. of tobacco fines were washed in 1 liter of distilled water to get rid of the tobacco solubles. The fines were then admixed with 1 N hydrochloric acid until the mixture had a pH of 1.35. The pH was checked on a Beckman pH machine and the curves were also plotted against the titration of distilled water.

After titration, the acid was washed off the fines with three 100 ml. portions of distilled water. The fines were redisbursed in 400 ml. of distilled water and a neutralization agent, as indicated in Table V, was added to bring the pH to the value shown in Table V. The slurry in each case thickened to a viscous mass which is evidence that the pectin had been solubilized. The slurry was cast at a thickness of 30–50 mils and dried to form sheets. The binders formed in this way were tested to determine their physical properties. These properties are shown in Table V below. The binder was used as the adhesive in a sandwich type reconstituted tobacco sheet.

TABLE V.—PROPERTIES OF BINDER FILM MADE BY ACID WASH PROCESS

Example	28	29	30
pH	7.1	7.0	7.1
Sheet Wt. gm./ft.	0.54	0.75	0.79
Neutralizing agent added	(2)	(2)	(2)
Integrator Count	936	864	864
Percent Moisture	10.8	11.0	12.1
Percent Elongation	18	19	19
Tensile kg./in.	2.72	2.93	2.88
Tensile Coefficient, kg./in. × gm./ft.	0.50	0.50	0.50
Work Coefficient, gm./sq. in.	4.91	5.49	5.47

(1) Conc. NH$_4$OH.
(2) 10% KOH.
(3) 10% NaOH.

In the table the terms have the following meanings:

(1) Integrator count is proportional to the area under the stress-strain curve recorded by an Instron Test Machine.

(2) Percent moisture=The percentage of moisture contained in the entire sheet (wet basis).

(3) Percent elongation=The percentage elongation at breakage, on an Instron Test Machine.

(4) Tensile kg./in.=The breaking strength of a 10 cm. test strip which is 1 inch wide; an average of 10 strips.

(5) Tensile coefficient=kg. per gm. of basis weight.

(6) Work coefficient gm./sq. in.=Work coefficient is proportional to the product of the tensile strength and the elongation.

Example 31

The following ingredients were mixed into 900 parts of hot water and heated for 3 hours at 85°–90° C., main-

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raising a pH of 7.0 by adding small amounts of aqueous ammonia.

	Parts
Mixed manufacturing byproducts and scrap tobacco	87.9
Diammonium orthophosphate (DAP)	5.0
Glycerin	5.0
Corn syrup	2.0
Potassium sorbate	0.14

The slurry was then refined in a one gallon capacity Waring Blender for 5 minutes. With this amount of refining, the fibrous portion of the composition was seen to be well dispersed and thoroughly interlocked when examined in a droplet at a 1:9 dilution. This examination was facilitated by the addition of a small amount of Congo Red dye.

The composition was cast on stainless steel panels with an eight inch angle knife set for wet film thicknesses of 0.020 to 0.045 inch, and dried on a steam table.

The wet strength of the films was determined on a Scott Serigraph IP-2 using 1 inch width strips. The center of each strip was wet with distilled water on both sides and allowed to become thoroughly wet through (30 seconds or one minute) before actuating the tensile mechanism. These films showed the extraordinary high wet film strength of 140 grams at a film weight of 9.5 grams per square foot. A high wet strength is highly desirable in the preparation of cigarette filter, and assures that the subsequent manufacturing performance of this material will exceed that of the finest grades of leaf tobacco. Such strength is uniquely obtainable in the presence of the liberated form of pectin described in this disclosure.

We claim:

Tobacco sheet material comprising finely divided tobacco bonded together by an adhesive derived from tobacco plant parts containing pectins having alkaline earth metal cross-links, said adhesive being prepared by the process comprising the steps of

(1) Contacting said tobacco plant parts with a treating solution containing a reagent comprising diammonium acid phosphate to destroy the alkaline earth metal cross-links,

(2) At least partially releasing the resulting tobacco pectins from the interstices of the treated tobacco plant parts;

(3) Depositing said tobacco pectins on the treated plant parts, and

(4) Recovering the resulting mixture.

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